

# ORGANIC CHEMISTRY OF BIVALENT SULFUR

VOLUME V

*by*

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1963

CHEMICAL PUBLISHING CO., INC.

*212 Fifth Avenue, New York, N. Y.*

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CHEMICAL PUBLISHING CO., INC.

New York

N. Y.

*Printed in the United States of America*

Chemistry  
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# Acknowledgments

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Publication of this work was supported in part by a grant from the Petroleum Research Fund administered by the American Chemical Society. Grateful acknowledgment is hereby made to the donors of said fund.

It is a pleasure to express my gratitude also to the Freeport Sulphur Company for their grant.

Special thanks are due to Dr. Jane Dick (Meyer) Clotworthy and to Dr. Edgar M. Faber, for invaluable assistance in the preparation of the manuscript and the reading of proofs; and to Norman Donaldson, for preparing the index.

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## CHAPTER 1

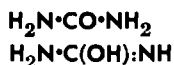
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# Thiourea

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### Introduction

As in other groups, the sulfur analog of urea shows many similarities to urea but also many striking differences.



Both urea and thiourea are tautomeric compounds. Reactions which involve the amino, or imino groups, are much the same in both, but in those that concern the  $\cdot\text{CO}\cdot$  and  $\cdot\text{CS}\cdot$  groups, the peculiarities of sulfur are evident; S-alkyl derivatives of pseudothiourea are readily formed.

### Occurrence

While urea is an abundant natural product, thiourea is rarely met with. It is produced by some fungi under special conditions.<sup>980</sup> The *D*-s-butyl derivative occurs in scurvy grass seeds.<sup>1306</sup> Benzylthiourea has been found in small amounts in dry papaya seed.<sup>374</sup>

### Formation

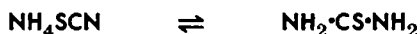
#### FROM AMMONIUM THIOCYANATE

One of the milestones in the development of organic chemistry was the isomerization of ammonium cyanate into urea:





This takes place rapidly at 100° and is virtually complete. The corresponding transformation of ammonium thiocyanate into thiourea, discovered by Reynolds, requires a much higher temperature and is by no means complete since the reaction is reversible: <sup>1052a</sup>



Reynolds heated ammonium thiocyanate to 170°, cooled it to 100°, added an equal weight of hot water and cooled further. Thiourea crystallized out. This reverted to ammonium thiocyanate when heated at 140°, showing that the reaction is reversible.<sup>223</sup> According to Claus, a mixture of alcohol, ammonia and carbon disulfide is evaporated until ammonium thiocyanate crystallizes out, and then heated over a free flame until the mass becomes blistery and fumes are evolved. Water is added to the cooled mass; thiourea is left undissolved.<sup>222d, 223</sup> If the mass is heated too high, guanidine thiocyanate and other products are formed.<sup>283, 1331</sup>

Pure thiourea melts at 184.5°. <sup>958</sup> This may hardly be considered a true melting point, since if it is cooled immediately and remelted, it melts at 149° indicating 70% conversion to ammonium thiocyanate.<sup>528</sup> Various possible reaction mechanisms have been proposed.<sup>1031</sup> Thiourea, crystallized from water, melts at 149°. <sup>335</sup> The freezing point curve of ammonium thiocyanate and thiourea shows a eutectic at 104.3°. <sup>536</sup> A later value is 100°. <sup>1333</sup> The two make a continuous series of solid solutions.

When ammonia and carbon disulfide react, the first product is ammonium thiocarbamate:



This loses hydrogen sulfide:



Below 120° ammonium thiocyanate is the chief product, but at 160° the yield of thiourea is high. This may be due to the transformation of ammonium thiocyanate into thiourea. As will appear later on, substituted thioureas are formed directly according to reaction 2. These two reactions are by no means the only ones that take place. The liberated hydrogen sulfide may combine

with ammonia and the ammonium sulfide thus formed, with carbon disulfide:



The ammonium trithiocarbonate breaks down, at high temperatures, into ammonium sulfide and carbon disulfide.<sup>461, 647, 930, 1428</sup> It is interesting that when the reaction of  $\text{NH}_3$  with  $\text{CS}_2$  is conducted at high pressures, guanidine is first formed as an intermediate and is then converted to melamine.<sup>747</sup>

The change of ammonium thiocyanate into thiourea and its reversal are both monomolecular.<sup>1337</sup> According to later observations the reverse reaction follows this law,<sup>337</sup> but the forward reaction is disturbed by side reactions.<sup>763</sup> The progress of the isomerization has been followed by viscosity measurements<sup>335</sup> and polarimetrically.<sup>991</sup> When ammonium thiocyanate is heated at  $170^\circ$  equilibrium is reached in an hour with 24.3% thiourea; at  $182^\circ$  it is reached in 30 minutes with 23.2%.<sup>1053</sup> In another study the mean heat of activation,  $140\text{--}80^\circ$ , was found to be 31,935 cal. for ammonium thiocyanate and 35,016 for thiourea. The percentage of thiourea formed and the heat of reaction were:<sup>691</sup>

|                | TU Formed | Cal. |                | TU Formed | Cal. |
|----------------|-----------|------|----------------|-----------|------|
| At $140^\circ$ | 28.1%     | 3271 | At $170^\circ$ | 23.1%     | 3074 |
| 150°           | 26.2%     | 3235 | 180°           | 21.8%     | —    |
| 160°           | 24.6%     | 3117 |                |           |      |

Approaching the equilibrium from both sides, closely agreeing data were obtained. Measurements were made with and without solvent:<sup>163</sup>

| Temp.       | No solvent | In propanol | In butanol |
|-------------|------------|-------------|------------|
| $132^\circ$ | 33.6%      | 19.3%       | 24.0%      |
| 156         | 26.8       | 23.5        | 28.5       |
| 182         | 22.2       | —           | —          |

The value for  $K$  for the equilibrium is 0.0254 at  $162^\circ$ , 0.0341 at  $178^\circ$  and 0.038 at  $184^\circ$ .<sup>1308</sup> In glycol solution the proportion of

thiourea is less at the greater dilution.<sup>1307</sup> An attempt was made to shift the equilibrium by the addition of salts that might combine with the thiourea and diminish its effective concentration. Potassium, cesium, and rubidium iodides were tried but all of them had the opposite effect.<sup>32</sup>

In working up the mixture obtained by heating ammonium thiocyanate, crystals melting at 144° and containing both thiourea and the starting material are obtained.<sup>186</sup> The melting point curve for mixtures of ammonium thiocyanate and thiourea was found to have an inflection indicating the presence of a compound,  $\text{NH}_4\text{SCN} \cdot 3\text{SC}(\text{NH}_2)_2$ , m. 144°. This can be crystallized from aqueous or alcoholic solutions of the melt.<sup>31, 32</sup> Other investigators arrived at the composition,  $\text{NH}_4\text{SCN} \cdot 4\text{SC}(\text{NH}_2)_2$ .<sup>1200</sup> The isomerizing of methyl- and dimethylamine thiocyanates, and benzylamine thiocyanate to the corresponding thioureas, together with activators for the change, have been investigated.<sup>508, 1101</sup>

The solubility of ammonium thiocyanate in 100 g. of water is 121.6 g. at 0° and 162 g. at 20°, whereas that of thiourea is only 9.18 g. at 13°. Following the original procedure of Reynolds, the separation of thiourea from the equilibrium mixture is practicable. Ammonium thiocyanate is heated at 170° for two hours. The melt is granulated and treated with a measured amount of cold water. Different proportions of water and different temperatures are recommended. On cooling, the thiourea comes out first, followed by the eutectic mixture. The solvent can be evaporated and the recovered ammonium thiocyanate containing some thiourea, reheated.<sup>316, 738, 760a, 768b, 961, 1165, 1305</sup> Liquid sulfur dioxide, in which thiourea is practically insoluble, is a convenient solvent for the separation.<sup>304, 738</sup>

Thiourea is not formed from thiophosgene and ammonia; ammonium thiocyanate is the product.<sup>1374f</sup>

#### FROM CYANAMIDE AND HYDROGEN SULFIDE

In 1872, Baumann got an almost quantitative yield of thiourea by bringing hydrogen sulfide and cyanamide together in absolute ether: <sup>68</sup>



A modification of this method was subsequently investigated and said to produce good yields.<sup>1214</sup> Ethyl sulfide and mercaptan did

not react with cyanamide, but thioacetic acid gave thiourea and acetylthiourea.<sup>68</sup> The reaction of thioacetic acid is vigorous.<sup>1024</sup> When cyanamide, in a solvent which is not miscible with water in all proportions (EtOAc or BuOH), is added to an aqueous solution of hydrogen sulfide so that two layers are formed, thiourea separates from the aqueous phase on cooling.<sup>1214</sup>

Cyanamide combines slowly with liquid hydrogen sulfide.<sup>602</sup>

The reaction of hydrogen sulfide with cyanamide goes satisfactorily in water. The cyanamide need not be prepared beforehand, but can be liberated from its calcium salt right in the reaction mixture. Thus an aqueous solution of calcium cyanamide is saturated with hydrogen sulfide and carbon dioxide passed in to precipitate calcium carbonate. This is filtered off and the solution concentrated to get the thiourea; <sup>464</sup> dilute sulfuric acid may be used with the calcium cyanamide solution and the calcium sulfate filtered off.<sup>1173</sup> The solution is made alkaline with ammonia and saturated with hydrogen sulfide.<sup>717</sup> The mother liquor from one run, already saturated with thiourea and calcium hydrosulfide may be used in the next run. Calcium cyanamide is added and hydrogen sulfide passed in, with stirring, to saturation. The solution is then heated to 90° for 15 minutes, filtered hot and cooled.<sup>1173</sup>

Hydrogen selenide and cyanamide unite to form selenourea,  $\text{SeC}(\text{NH}_2)_2$ , m. 213° dec.<sup>40</sup> A 25% aqueous solution of cyanamide containing a trace of hydrochloric acid is saturated with hydrogen selenide, cooled to 0°, and the product filtered off.<sup>333</sup>

A review of the preparation and biological properties of thioureas has been made.<sup>1152</sup>

### The Manufacture of Thiourea

For many years thiourea has been manufactured in Germany from ammonium thiocyanate and exported to all parts of the world. Its commercial production was begun in the United States in 1940.<sup>15, 760b</sup>

The usual starting material is calcium cyanamide. There are numerous patents covering specific ways of bringing the reactants together and of isolating the thiourea. A dilute aqueous solution of cyanamide is treated at elevated temperature with hydrogen sulfide. Solids and acids or alkalis may be present.<sup>693</sup> Cyanamide, in aqueous solution, with a minor amount of ammonium

hydrosulfide, is treated with hydrogen sulfide.<sup>557</sup> Calcium cyanamide is treated with carbon dioxide and hydrogen sulfide at ordinary or raised pressure with cooling,<sup>290</sup> preferably at 0°. <sup>1154</sup> Carbon dioxide, under about 5 atmospheres pressure, is led into a closed vessel at a temperature above 40° to react with a mixture of calcium cyanamide and calcium sulfide,<sup>1216, 1217, 1290</sup> or the calcium cyanamide, may be ground with water and hydrogen sulfide passed in at 50° or above.<sup>1217</sup> Barium sulfide may be substituted for the calcium sulfide.<sup>442</sup> A slurry of calcium cyanamide in water is treated with carbon dioxide or sulfur dioxide and then saturated with hydrogen sulfide.<sup>440</sup>

A suspension of calcium cyanamide in water is treated with hydrogen sulfide and the resulting calcium sulfide decomposed at 45 to 50° by addition of  $\text{H}_2\text{SO}_4$ ,  $\text{H}_3\text{PO}_4$  or  $\text{H}_2\text{CO}_3$  in the catalytic presence of  $\text{PhNH}_2$ . A conversion of 98.5 to 99.5% is claimed, and also that the product is not contaminated with by-products.<sup>787</sup>

The hydrogen sulfide contained as an impurity in hydrocarbon gases may be utilized for making thiourea by bringing the mixture into contact with calcium cyanamide.<sup>817</sup> Any iron in the calcium cyanamide may be eliminated by a preliminary treatment with hydrogen sulfide.<sup>5</sup> Thiourea may be produced by the reaction of hydrogen sulfide on a heavy metal cyanamide,<sup>486</sup> or heating sodium sulfide, calcium cyanamide and calcium chloride in aqueous solution at 75–80°. <sup>1310</sup>

Thiourea labeled with either sulfur 35 or carbon 14 has been prepared utilizing similar syntheses to the above-mentioned ones.<sup>99, 866</sup>

A recent method of preparing thiourea involves the use of arsenic sulfide ore. A water suspension of finely ground arsenic sulfide ore, containing a small amount of sulfur (5%) is mixed with the proper amount of calcium cyanamide, the ratio of nitrogen in the latter to the sulfur in the ore being 1:1. After heating at 90° for one-half to one hour, filtering, and neutralizing the filtrate with sulfuric acid, the precipitated sulfide is filtered off. The thiourea filtrate is evaporated to initial crystallization and left to crystallize. After further purification by recrystallization the yield of thiourea is 80–90%. <sup>1060</sup>

Calcium cyanamide, in aqueous solution, is reacted with ammonium sulfide and sulfur,<sup>1360</sup> or with the reaction products of

ammonia and phosphorus pentasulfide.<sup>557</sup> A mixture of calcium cyanamide, ammonium sulfide and ammonium salts of acids, such as carbonic, oxalic or sulfuric, the calcium salts of which are insoluble in water, is stirred into water.<sup>601, 631, 899</sup> Calcium cyanamide is reacted with hydrogen sulfide, under pressure, with stirring, and at temperatures below 100°, in the absence of any liquid or in the presence of an amount of water or organic liquid sufficient to make a paste.<sup>609, 640</sup> Calcium cyanamide is added in portions to hydrogen sulfide which is always maintained in excess.<sup>241</sup>

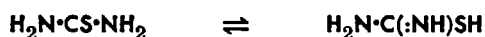
Thiourea is produced by treating a mixture of calcium cyanamide and crystalline alkali sulfates at ordinary or raised temperatures with hydrogen sulfide, or from calcium cyanamide, soluble alkali hydrosulfides and sulfur at temperatures below 150°.<sup>548</sup>

In a cyclic process of manufacture, thiourea is synthesized in a solution of thiourea. The resulting solution is chilled and thiourea filtered off after which the mother liquor is recycled.<sup>1067a</sup>

The finely-divided sulfur recovered from coal gas is converted by ammonia and hydrocyanic acid into thiocyanates and thiourea.<sup>658</sup>

### Structure

There has been much discussion as to the structure of thiourea and its derivatives. Two structures are possible:



The first of these is called the normal, and the other, the pseudo or iso form. Some chemists have argued for the one and some for the other. It is generally agreed that thiourea is tautomeric and may react as if it had either structure. The same may be said for substituted thioureas except, of course, the tetra- $\text{R}_2\text{N}\cdot\text{CS}\cdot\text{NR}_2$ . This view was held by Foerster.<sup>409</sup> Rathke wrote the formula of diphenylthiourea  $\text{PhNH}\cdot\text{C}(\text{:NPh})\text{SH}$ , since it gives a silver salt, while the ethyl derivative  $\text{PhNH}\cdot\text{C}(\text{:NPh})\text{SEt}$  does not.<sup>1037b</sup> After reviewing all the evidence, Dixon and Taylor concluded that thiourea and its N-alkyl and acyl derivatives have the "normal" structure  $\text{:N}\cdot\text{CS}\cdot\text{N:}$  as long as they remain in the static condition, but may react as if they had the other form.<sup>308b</sup>

A recent study has been made of the equilibria between these

two forms with the suggestion of a third form having a double minus charge.<sup>507</sup>  $\text{SC}(\text{NHPH})_2$  showed tautomeric equilibrium with the thienol form,  $\text{HSC}(:\text{NPh})\text{NHPH}$ , in  $\text{C}_5\text{H}_5\text{N}$ .<sup>754</sup>

Parachor data agree with the structure  $\text{SC}(\text{NH}_2)_2$ .<sup>1044</sup> Conductivity, cryoscopic and hydrogen ion determinations show that thiourea and its alkyl derivatives do not exist in dilute neutral solutions in the ionizable form,  $\text{HN}:\text{C}(\text{NH}_2)\text{SH}$ . Only two forms

are possible:  $\text{SC}(\text{NH}_2)_2$  and  $\text{HN}:\text{C} \begin{array}{c} \text{NH}_3 \\ | \\ \text{S} \end{array}$ .<sup>251, 507</sup> An earlier

study by Werner<sup>1374d</sup> of oxidation and alkylation by diazomethane pointed to an equilibrium between  $\text{HN}:\text{C}(\text{NH}_2)\text{SH}$  and

$\text{HN}:\text{C} \begin{array}{c} \text{NH}_3 \\ | \\ \text{S} \end{array}$ . Oxidation to the disulfide indicates the structure

$\text{NH}_2\cdot\text{C}(:\text{NH})\text{SH}$ .<sup>884, 1251</sup> Potentiometric studies of the constitution of thiourea and phenylthiourea have been made by following the  $\text{NaOH}$ -titration curves in dilute aqueous solutions.<sup>509</sup> The structures of thioureas and thiuronium salts have been clarified somewhat by studying some reactions of 1,1,3-trimethyl-2-thiourea.<sup>809</sup>

A comparison of the absorption spectra of thiourea and of a number of its tri- and tetra- derivatives led to the conclusion that thiourea and its trimethyl derivative have the iso-structure,  $\text{H}_2\text{N}\cdot\text{C}(:\text{NH})\text{SH}$ .<sup>1064</sup> Thiourea has a greater power of general ultraviolet absorption than can be attributed to the presence of only one unsaturated center.<sup>845, 885</sup> From the Raman spectra, it is concluded that thiourea exists in the amphoteric-ion form. A strong  $\text{C}=\text{N}$  frequency is found only in the tetra methylisothiourea,  $\text{MeN}:\text{C}(\text{SMe})\cdot\text{NMe}_2$ .<sup>749</sup> The Raman spectra of thiourea indicate that it possesses a two-fold axis symmetry.<sup>354</sup> Other absorption spectra studies have been made.<sup>44, 1245</sup> Dipole moment values indicate that thiourea and urea are resonance hybrids with a 20–30% contribution from the forms with a separation of charges. Their structures are not essentially different from those of simple amides.<sup>775</sup> An osmometric study of thiourea using a rush membrane has thrown some light on the mesohydric tautomerism of thiourea.<sup>1284</sup> The resonance energy of thiourea has been calculated to be 27 kilocalories per mole.<sup>1257</sup> The diamagnetic susceptibility and the ultraviolet absorption also indicate that thiourea is a resonance hybrid.<sup>228, 229</sup> The dielectric con-

stants of solutions of thiourea and of some of its derivatives in water<sup>291</sup> and in dioxane<sup>82</sup> indicate that thiourea exists to a large extent as the zwitter ion,  $\text{H}_2\text{N}^+:\text{C}(\text{NH}_2)\text{S}^-$ . The substituted thioureas have this form to a lesser extent.<sup>82</sup> In a long study of this question, Lecher and coworkers favored the zwitter ion for a time but finally concluded that it is impossible, and returned to the structure  $\text{SC}(\text{NH}_2)_2$ .<sup>804, 805, 806, 807</sup> The dielectric constant in aqueous solution is 4.0 compared with 3.3 for urea.<sup>189</sup> The relation between the structure of formamidine disulfide to thiourea has been shown.<sup>1101</sup>

The unit of structure of thiourea contains four molecules, a 5.47, b 7.64 and c 8.5 A.U.<sup>284</sup> Other values are 5.50, 7.68 and 8.57 A.U.<sup>547</sup> The density is 1.408.<sup>284</sup> All of the atoms in the molecule lie practically in one plane. The distances C-N and N-N are 1.35 and 2.18 A.U., respectively, as compared to 1.33 and 2.24 for urea. The C-S distance is 1.64.<sup>1408</sup> Thiourea is rhombic.<sup>565</sup>

Thiourea has been examined under the polarizing microscope for polymorphism.<sup>1118</sup> In the molten condition three forms appear to be present.<sup>901</sup> The thaw melting point diagram for urea-thiourea binary system has been determined.<sup>1054</sup> High pressure causes a transition of the solid form.<sup>142</sup> The change from the unstable to the stable form starts at many points.<sup>932</sup> The crystal structure of ethylene thiourea has been analyzed in detail; a length of 1.59 A.U. is deduced from  $\text{C}=\text{S}$ .<sup>1379</sup> The magnetic rotation of the plane of polarization of light for thiourea has been compared with that of a number of other substances.<sup>10</sup> Thiourea exhibits crossed axial plane dispersion near 3780 A.U. in ultra-violet.<sup>156</sup> Its infrared absorption has been compared with those of a number of its derivatives.<sup>167, 893</sup>

The solubilities of thiourea in water, pyridine and pyridine hydrate at 25° are 9.10, 12.50 and 41.20 g. per 100 g. of solvent.<sup>279</sup> The solubilities in water, methanol and ethanol are in Table 1.4.<sup>1176</sup>

TABLE 1.1  
*Solubility of Thiourea in 100 g. of Solvent*

| Temp. | Water   | Methanol | Ethanol |
|-------|---------|----------|---------|
| 20°   | 13.70 g | 11.97 g  | 3.71 g  |
| 25    | 17.12   | 13.48    | 4.27    |
| 75    | 117.3   | 40.22    | 12.52   |



Rates of decomposition of thiourea in water were measured at 5 to 960 millimolar concentrations. The reaction was first order and the rate was independent of acid concentration over a wide range and did not depend on ionic strength.<sup>1168</sup>

The base strength of urea and thiourea in methanol has been determined. The value of  $K_b$  for thiourea is  $5.46 \pm 0.1 \times 10^{-2}$ , and for urea is  $4.37 \pm 0.2 \times 10^{-4}$ ; the corresponding hydrolysis constants in water are 9.0 and 0.67.<sup>995</sup>

The reaction of i-cholesteryl methyl ether, H ion, and thiourea to form a cholesterylisothiuronium salt is first order in each reactant. The 3rd-order rate constant for this reaction has been measured as well as the 2nd-order rate.<sup>994</sup>

The heat of combustion has been determined.<sup>882</sup> Its hardness and flow pressure have been compared with those of other substances.<sup>779</sup> It gives peculiar effects when evaporated on a glass plate.<sup>451</sup> The wetting of a mercury surface by a solution of thiourea is attributed to the affinity of the sulfur atom for the metal.<sup>204</sup> The swelling of cellulose, starch and gelatine in water is increased by the presence of thiourea.<sup>697</sup> The influence of temperature on the statics and kinetics of the cellular penetration of urea and thiourea into baking yeast has been determined.<sup>824</sup>

The polarographic properties of sodium N-(p-Chlorophenyl) diazothiurea have been studied.<sup>826</sup> The specific conductance of a saturated solution of thiocarbanilide in liquid  $H_2S$  has been compared with other thio organics.<sup>1030</sup> Substituted thiourea derivatives of the type, 1-(2-carboxyethyl)-3-(p-nitrophenyl) thiourea, are very sweet to the taste, as much as 350 times as sweet as sucrose.<sup>1000</sup>

## Reactions

### COMPLEXES WITH METAL SALTS

Thiourea is remarkable for the number and variety of the addition compounds which it forms with salts of metals, all the way from potassium in the first group to platinum in the eighth. Its alkyl derivatives share this property. The combinations of 78 inorganic ions with 60 substituted thioureas have been studied. Many of them serve as sensitive color tests for the ions.<sup>981, 1413</sup> Many addition compounds in solution have been detected by physical measurements.<sup>63</sup> The relative stability of some metal complexes of thiourea and substituted thioureas has been investi-

gated by means of a potentiometric procedure.<sup>1353</sup> Thiourea forms large complex metal cations which can be precipitated by large anions.<sup>1412</sup>

Ammonia gives the compounds,  $\text{SC}(\text{NH}_2)_2\text{NH}_3$  and  $\text{SC}(\text{NH}_2)_2\cdot 3\text{NH}_3$ .<sup>664</sup> Four molecules of thiourea combine with one of ammonium chloride, bromide or iodide, or with one of tetraethylammonium bromide or iodide.<sup>1052c</sup> There are also addition compounds with complicated organic bases.<sup>1193</sup> It combines in the 4:1 ratio with potassium,<sup>32, 894, 1074, 1374c</sup> rubidium<sup>32, 35</sup> and cesium<sup>32</sup> iodides. Thiourea, allyl- and acetyl-thiourea give compounds with calcium chloride and iodide.<sup>487, 1075</sup> Thiourea combines with all manner of silver salts.<sup>68, 222c, 365, 384, 778, 795, 867a, 1037b, 1052a, 1052c, 1074, 1244, 1350</sup> Silver bromide<sup>365</sup> and silver metal dissolve in an aqueous solution of thiourea.<sup>1010</sup> There are numerous compounds with copper salts.<sup>239, 279, 750, 751, 778, 825, 909, 920, 1024, 1037b, 1074, 1076, 1253, 1350, 1352</sup>

Complexes containing thiourea and mercuric chloride, cyanide, nitrite<sup>222c, 778, 825, 867b, 948a, 1014, 1040, 1043, 1052a, 1075</sup> and sulfate,<sup>1075</sup> iodide<sup>867b, 1075</sup> and thiocyanate<sup>1075</sup> are known, so are a number with gold<sup>912, 1010, 1024, 1052a</sup> and platinum salts.<sup>36, 481, 491, 778, 948a, 1014, 1024, 1052a, 1091, 1162, 1234, 1265</sup> Palladium,<sup>366, 491, 778, 1014, 1092</sup> lead,<sup>825, 862a, 1075, 1350</sup> zinc,<sup>867b, 1075, 1350</sup> cobalt,<sup>1014, 1075</sup> cadmium,<sup>862a, 867b, 1075, 1350</sup> manganese,<sup>1075</sup> tin,<sup>867b, 1350</sup> iron,<sup>825, 1075, 1350</sup> antimony,<sup>848, 1314</sup> bismuth,<sup>1314</sup> chromium,<sup>862a, 1003</sup> nickel,<sup>825, 1075, 1349b, 1350</sup> arsenic,<sup>825, 1349b</sup> rhodium,<sup>819</sup> iridium,<sup>501, 799</sup> osmium,<sup>213b, 1110a</sup> and thallium<sup>475</sup> salts combine with thiourea. The osmium and ruthenium complexes are highly colored and serve for the detection of these elements.<sup>213b, 460, 1197, 1316, 1400, 1401</sup> Thiourea makes possible the precipitation of cadmium in the presence of zinc<sup>127, 285, 861b, 862a</sup> and the separation of thallium from most other ions.<sup>862a, 1024, 1074</sup> Lead can be precipitated in the presence of other metals.<sup>332, 825, 862b</sup> As little as 0.02 gamma of lead can be detected by the formation of the complex  $2\text{Pb}(\text{NO}_3)_2 \cdot 11\text{CS}(\text{NH}_2)_2$ .<sup>861b</sup> Copper and cadmium can be determined colorimetrically with Reinecke's salt and thiourea.<sup>861b</sup> The complexes with bismuth salts are colored and serve for the detection of the bismuth ion<sup>329, 330, 331, 332, 496, 571, 670, 825, 848, 861a, 1001, 1186, 1314, 1426</sup> and its colorimetric estimation.<sup>861a, 862a, 1001</sup> The crystal forms of  $\text{BiCl}_3 \cdot 3\text{CS}(\text{NH}_2)_2$  and  $\text{BiCo}(\text{CN})_6 \cdot 6\text{CS}(\text{NH}_2)_2$  have been determined.<sup>500</sup> The complex salt  $\text{Cu}_3\text{Fe}(\text{CN})_6 \cdot 3\text{CS}(\text{NH}_2)_2 \cdot 2\text{H}_2\text{O}$  is a sensitive test for copper or for ferrocyanide.<sup>1253</sup> Thiourea

precipitates red selenium from a hydrochloric acid solution.<sup>285, 376</sup> It serves to detect one part of tellurium in 500,000 of solution.<sup>383a</sup>

Sulfathiourea forms a complex with copper.<sup>313</sup> Allylthiourea forms complexes with silver chloride, bromide, iodide<sup>1170</sup> and nitrate<sup>439a</sup> and with mercuric chloride, cyanide<sup>439a</sup> and iodide<sup>410</sup> and with cuprous chloride.<sup>439a</sup> It gives characteristic tests with cadmium ions.<sup>512</sup> Ethylenethiourea combines with mercury<sup>569e, 920, 1130</sup> silver,<sup>569e, 920, 1130</sup> cadmium,<sup>920</sup> lead,<sup>920</sup> copper,<sup>920</sup> palladium,<sup>920</sup> platinum,<sup>569e, 1130</sup> and gold<sup>569e, 920, 1130</sup> salts. The gold and copper compounds have been tested therapeutically.<sup>309</sup> The 1,2-dimethyl forms double salts with mercury and platinum chlorides.<sup>1416</sup> Thiosemicarbazide and several of its derivatives form complexes with silver nitrate.<sup>530</sup> Thiourea and sodium nitroprusside give complicated salts.<sup>176</sup> Guanidylthiourea carbonate is a reagent for silver ions.<sup>256</sup> Silicon tetrabromide gives the addition compound,  $\text{SiBr}_4 \cdot 8\text{CS}(\text{NH}_2)_2$ .<sup>1052c</sup> The use of thiourea in analysis is discussed by Yoe.<sup>1414</sup>

Conversely, the presence of thiourea may be established by the formation of complexes with metal salts.<sup>285, 390, 1253</sup> Thiourea can be taken out of a solution by adding cadmium sulfate, boiling and filtering.<sup>1390</sup>

Methyl-, ethyl-, diethyl- and triethyl-thioureas give complexes with silver, mercury, platinum and palladium salts.<sup>778, 1101</sup> Disoundecylthiourea forms double salts with mercury and platinum chlorides.<sup>1022</sup> The methyl-, ethyl- and allyl- hydroxythioureas,  $\text{RNH} \cdot \text{CS} \cdot \text{NH}(\text{OH})$  and  $\text{RNH} \cdot \text{CS} \cdot \text{N}(\text{OH})\text{R}'$  give compounds with silver, mercury and gold salts.<sup>736</sup>

Phenylthiourea gives complexes with mercuric salts<sup>1042</sup> and with bismuth chloride.<sup>1314</sup> It interferes with the Volhard titration of thiocyanates.<sup>785</sup> *s*-Diphenylthiourea combines with bismuth chloride<sup>1314</sup> and gives a blue-green color with 0.3 gamma of ruthenium ion per cubic centimeter.<sup>1400</sup> With osmium the color is red.<sup>1400</sup> The *N*-hydroxy derivatives,  $\text{PhN}(\text{OH}) \cdot \text{CS} \cdot \text{NH}_2$ ,  $\text{PhN}(\text{OH}) \cdot \text{CS} \cdot \text{NHPh}$ ,  $\text{PhN}(\text{OH}) \cdot \text{CS} \cdot \text{NHC}_3\text{H}_5$ , give stable precipitates with many metal ions.<sup>1177</sup> 4,4'-Diacetylaminodiphenylthiourea is a sensitive reagent for lead ions and 4,4'-dimethyl-3,3'-dinitrodiphenylthiourea for copper and silver.<sup>1276</sup>

#### ADDUCTS WITH HYDROCARBONS

Thiourea forms crystalline complexes with many organic compounds at room temperature; in many cases, 3 moles thiourea to

1 mole compound. The complexes are stable only in crystalline form and dissociate at higher temperatures or in solution.<sup>14, 1128, 1132, 1278</sup> The most important are the complexes with hydrocarbons which closely resemble those formed by urea, the distinguishing difference being the tendency of the thioureas to combine with branched chain hydrocarbons, whereas the ureas form complexes with straight chain hydrocarbons.<sup>14, 66, 482, 843, 1045</sup> This fact has been utilized in the separation of normal hydrocarbons from branched chain hydrocarbons<sup>66, 396, 721</sup> and is discussed later under urea. The formation of adducts, however, is limited to hydrocarbons containing more than twelve carbon atoms.<sup>854</sup>

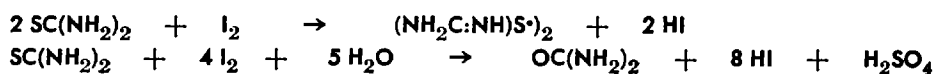
In the thiourea complexes the cell is rhombohedral, but the arrangement of the thiourea molecules is similar to that of the urea molecules in the urea complexes.<sup>1204</sup>

#### DETECTION AND ESTIMATION OF THIOUREA

Qualitative and quantitative analytical methods for thiourea have been summarized.<sup>285, 383b</sup> The significance of thiourea in plant physiology makes its estimation important.<sup>1273</sup> In fungus extracts it can be determined spectroscopically.<sup>1419</sup> An acid solution of copper sulfate detects certain substituted thioureas.<sup>129, 353, 770</sup>

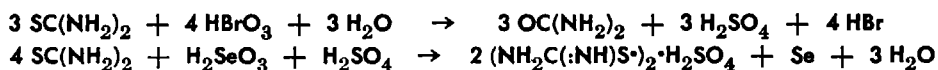
Thiourea is estimated by titration with oxidising solutions,<sup>1018, 1053</sup> permanganate, chloramine-T, cerium sulfate or chromate.<sup>280</sup> When NaClO or HClO<sub>4</sub> and HNO<sub>3</sub> are used as an oxidising agent, the sulfur of thiourea is determined as the sulfate.<sup>684, 1077</sup> In acid solution, permanganate stops at the disulfide, but in neutral solution takes the sulfur all the way to sulfuric acid.<sup>867c</sup> In the case of tri- and tetramethylenethiourea, values are often too low when oxidised with nitric acid due to the formation of volatile COS. When the fumes from this oxidation are passed over platinum catalysts in a combustion tube at 500°, the sulfur of the COS can be determined.<sup>1237a</sup>

Iodine oxidises thiourea to the disulfide in neutral as well as in acid solution.<sup>231, 1374d</sup> Eight times as much iodine is required in alkaline solution as in acid:



Nitric acid, copper, and mercury salts interfere in the acid titra-

tion.<sup>861c, 1201</sup> With bromate and selenious acid the reactions are: <sup>677, 1266, 1373</sup>



Thiourea serves for the titrimetric determination of oxidising agents such as lead peroxide, manganese dioxide, hydrogen peroxide, chromate <sup>861a, 862b</sup> and persulfates.<sup>862b</sup>

Thiourea may be titrated with silver nitrate <sup>1, 260, 956, 1331</sup> or mercuric nitrate.<sup>719, 1390</sup> It can be determined colorimetrically by a reagent prepared from sodium nitroprusside, hydroxylamine and bromine.<sup>499, 273</sup> Improvements in the preparation and use of this reagent have been described.<sup>205a</sup> A color reaction with potassium ferrocyanide detects thiourea down to 1 part in 20,000.<sup>1109</sup> The thiourea may be converted to thiocyanate ion by nitrous acid. Ferric chloride is added and the ferric thiocyanate determined colorimetrically.<sup>180</sup> A new test using  $\text{K}_2\text{HgI}_4$  has been devised.<sup>554</sup>

A more recent color reagent for the detection of thioureas consists of separate solutions of o-toluidine, cupric chloride, and sodium acetate. A drop of each on a spot plate to which is added a drop of thiourea solution produces an intense blue color.<sup>955</sup> The colorimetric detection of  $\alpha$ -naphthylthiourea (ANTU) has been studied in connection with pharmacologic investigations.<sup>339</sup> Thiourea may be determined potentiometrically with  $\text{ICl}$ .<sup>216</sup>

Infrared <sup>790, 893</sup> and ultraviolet studies <sup>375</sup> have been made of the thiourea group. The polarographic behavior of thiourea and its derivatives has been observed.<sup>391, 668</sup> Thiourea and its derivatives have been analyzed by chromatographic methods.<sup>339, 573, 728, 733</sup> The analysis of the thioureas obtained from mustard oils has been discussed.<sup>375, 1117</sup> The thiourea present in foods has been determined.<sup>483, 1397</sup>

#### ADDITION OF ACIDS

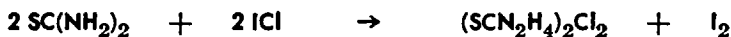
Thiourea combines with acids but the products appear to be addition compounds rather than salts. Its aqueous solution contains no hydroxyl ions; there is no heat of neutralization when an acid is added. It does not affect the optical rotation of tar-

taric acid.<sup>497</sup> The conductivity of its solution is extremely low.<sup>1291</sup> It is a very weak base.<sup>1344, 1345</sup>

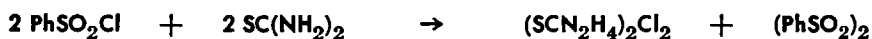
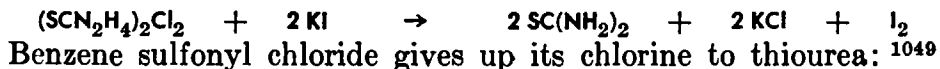
The hydrochloride,  $\text{SC}(\text{NH}_2)_2 \cdot \text{HCl}$ , has been prepared by saturating an alcoholic solution of thiourea with hydrogen chloride and adding ether,<sup>528</sup> or by dissolving thiourea in warm concentrated hydrochloric acid, saturating this solution with hydrogen chloride and cooling. The crystals are washed with chloroform; they are stable in dry air and melt at  $136-7^\circ$ .<sup>305, 305e, 1244</sup> It is completely hydrolyzed when dissolved in water.<sup>1402</sup> Gaseous hydrogen chloride is absorbed by thiourea.<sup>528</sup> The sulfate,  $(\text{NH}_2)_2\text{CS} \cdot \text{H}_2\text{SO}_4$ , melts at  $9^\circ$ , the oxalate,  $(\text{NH}_2)_2\text{CS} \cdot (\text{COOH})_2$ , at  $73-4^\circ$ . The nitrate explodes on drying unless prepared with care.<sup>305a</sup> Thiourea nitrate can be transformed to nitrothiourea by treatment with acetanhydride in trichloroethane.<sup>983</sup> Thiourea forms addition compounds with maleic and cinnamic acids.<sup>1090</sup> Carboxylic acid esters of S-alkylisothioureas are prepared by treating  $\text{CH}_2\text{ClCH}_2\text{OH}$  esters of higher carboxylic acids with thiourea.<sup>685</sup>

#### ADDITION REACTIONS

When bromine is added to a concentrated alcoholic solution of thiourea an addition product,  $\text{C}_2\text{H}_8\text{N}_4\text{S}_2\text{Br}_2$ , crystallizes out. The corresponding chlorine compound,  $(\text{SCN}_2\text{H}_4)_2\text{Cl}_2$ , was similarly obtained.<sup>222b, 222a</sup> The required chlorine may be abstracted by the thiourea from trichloromethane sulfonyl chloride, methane sulfonyl chloride or iodine chloride:



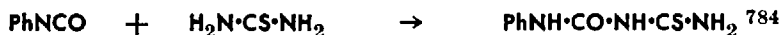
The iodine compound is formed when thiourea and iodine are rubbed together in a mortar. The chlorine compound liberates iodine from the potassium iodide:<sup>851</sup>



An aqueous solution of thiourea and iodine contains the addition compound in equilibrium with its constituents:<sup>874</sup>



Thiourea combines with an isocyanate:



With an acylisocyanate the product is an acylthiobiuret:



The reaction takes place slowly in hot benzene.

Thiourea adds to the triple bond of phenylpropionic acid:



The product decomposes into cyanamide and a mercaptan.<sup>402</sup> Thiourea reacts with dichloroacetic acid to give a thiohydantoin,<sup>305b</sup> and with oxalacetic acid to form thio-orotic acid.<sup>38</sup> It forms addition compounds with quaternary ammonium iodides.<sup>1193</sup> Bis (quaternary alkylammonium ethyl) sulfide salts have been prepared from thiourea and bifunctional tertiary amine salts.<sup>382</sup> The kinetics of the heterogeneous ammoniation of thiourea has been studied.<sup>1362</sup> Heterocyclic compounds which contain a saturated side chain in the alpha position readily form addition compounds with thiourea, but in the case of an alpha unsaturated side chain no adducts are formed.<sup>1371</sup>

When thiourea is combined with cyanamide in the presence of mineral acids, a thiodiformamidine is formed, m. 146.<sup>191</sup> The reaction of thiourea with  $\beta$ -propiolactone in an aqueous medium produces the  $\beta$ -isothioureia derivative of propionic acid,  $\text{H}_2\text{NC}(\text{:NH})\text{SCH}_2\text{CH}_2\text{COOH}$ , in high yield. This reaction is an example of the preparation of  $\beta$ -substituted carboxylic acids from saturated aliphatic  $\beta$ -lactones and compounds having a  $-\text{C}(\text{:S})\text{NH}$  linkage.<sup>477</sup> Thiourea reacts with benzyl halo nitriles in acetone to give thiazoles and thiadiazines.<sup>199</sup> Thiourea and ethyl oxalate form  $\text{SC}(\text{NH}_2)_2\cdot(\text{CO}_2\text{Et})_2$  which is decomposed into its constituents by heat or by hot water. On warming it with aldehydeammonia, diethylidenethiourea is produced.<sup>948b</sup>

Of special interest is the Traube synthesis of purines utilizing thiourea and compounds like  $\beta$ -carbonyl acetates and ethylcyanoacetate.<sup>1288</sup> Several complex pyrimidines have been prepared from thiourea.<sup>93</sup> Lithium thiocyanate is formed from thiourea and lithium carbonate.<sup>1277</sup> Diketene reacts with thiourea to give

6-methyl-2-thiouracil.<sup>783</sup> It combines with sulfur monochloride to form  $\alpha,\alpha'$ -dithiodiformamidine.<sup>1096</sup> Spiro derivatives of thio-barbituric acid may be prepared by adding  $(\text{CH}=\text{CH}_2)_2$  to dialkyl alkylidene-malonates and condensing the intermediate esters formed with thiourea.<sup>242</sup>

The irradiation of thiourea by gamma rays, X-rays or electrons, liberates sulfur via a chain reaction. After a characteristic induction period the sulfur yield rises to characteristic maximums and plateaus depending on the dose rate.<sup>266</sup>

### ISOTHIURONIUM SALTS

#### General

The addition of alkyl halides is one of the most characteristic and useful reactions of thiourea. The addition takes place readily: <sup>222b, 224</sup>



The reaction takes place readily and is quite general,<sup>41</sup> even with high ones such as cetyl,<sup>1231</sup> cycloaliphatic<sup>42</sup> and tertiary,<sup>39, 42, 1150</sup> dihalides,<sup>12b, 95, 303, 699</sup> sugar derivatives,<sup>1144</sup> hydroxyhalides,<sup>227, 973, 1114</sup> halogenated acids<sup>12a, 1041, 1331</sup> and alpha chloromethyl ethers,  $\text{ROCH}_2\text{Cl}$ .<sup>1222a</sup> The rate of the addition depends on the halide and on the solvent. In acetone methyl iodide adds to thiourea forty-three times as fast as ethyl iodide. The reaction follows the bimolecular law closely. Values of K for measurements at 25° with time in hours are given for several solvents and for several substituted thioureas (Table 2.4).

TABLE 2.1

*Values of K for Addition of MeI to Thioureas*

| Thiourea  | EtOH | MeOH | i-BuOH | AcOEt | AcMe | PhNO <sub>2</sub> |
|---|------|------|--------|-------|------|-------------------|
| $\text{H}_2\text{N}\cdot\text{CS}\cdot\text{NH}_2$            | 13.6 | —    | —      | —     | 60   | —                 |
| $\text{MeNH}\cdot\text{CS}\cdot\text{NH}_2$                   | 11.6 | —    | 6.0    | —     | 14.0 | —                 |
| $\text{C}_6\text{H}_5\text{NH}\cdot\text{CS}\cdot\text{NH}_2$ | 8.7  | 8.0  | 5.2    | 10.0  | 29.8 | 13.2              |
| $\text{PhNH}\cdot\text{CS}\cdot\text{NH}_2$                   | 8.6  | —    | —      | —     | 22.4 | —                 |
| $\text{Ph}(\text{Me})\text{N}\cdot\text{CS}\cdot\text{NH}_2$  | 4.8  | 4.4  | —      | —     | —    | —                 |
| $\text{EtNH}\cdot\text{CS}\cdot\text{NHC}_2\text{H}_5$        | 2.9  | 3.2  | —      | —     | —    | —                 |
| $\text{C}_6\text{H}_5\text{NH}\cdot\text{CS}\cdot\text{NHPh}$ | 3.4  | 2.8  | 2.0    | —     | 4.9  | 3.7               |



The rate in acetone is usually several times what it is in ethanol in which it is slightly greater than in methanol. Substituting methyl, allyl or phenyl in one of the  $-\text{NH}_2$  groups lowers the rate. A second substituent reduces the rate still more, particularly if it is on the other nitrogen atom.<sup>471, 472</sup> The addition of 4% of water to the ethyl alcohol solution does not affect the rate of addition of ethyl iodide; 20% raises it somewhat.<sup>472</sup>

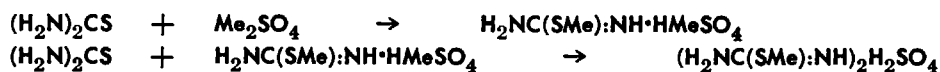
The products of the addition,  $\text{RSC}(:\text{NH})\text{NH}_2\cdot\text{HX}$ , are known as S-alkylisothiuronium salts. They are stable neutral salts. Thiourea which is scarcely a base at all becomes a strong base when a hydrogen of the  $-\text{SH}$  of the pseudo form,  $\text{HSC}(:\text{NH})\text{NH}_2$ , of thiourea is substituted by an alkyl. The addition of alkali to one of these salts liberates the base,  $\text{RSC}(:\text{NH})\text{NH}_2$ . Instead of the halides, alkyl sulfates, nitrates and thiocyanates may be used.<sup>789, 1274</sup> Several alkylisothiuronium sulfates and nitrates have been nitrated to give nitro thiopseudoureas.<sup>404</sup>

### Preparation

To prepare the isothiuronium salts the reactants are brought together in a flask fitted with a reflux condenser. Some solvent is added, water for the lower halides and alcohol for the higher less soluble ones. The reaction may take place spontaneously or may have to be started by heating. It is sometimes necessary to moderate it by cooling. Usually the mixture is heated a while to complete the reaction. For the alkyl bromides from heptyl to octadecyl, 0.5 mole of the bromide and 0.5 mole of thiourea and 250 cc. of alcohol are heated on a steam cone for 3 hours. The yields are high.<sup>1309</sup> The salts may be recrystallized and many have satisfactory melting points; that from n-propyl bromide melts at  $60^\circ$ , from i-butyl at  $96^\circ$ , from isoamyl at  $84^\circ$ <sup>1381</sup> and from allyl at  $85^\circ$ .<sup>1374a</sup>

The use of the esters of p-toluene-sulfonic acids as alkylating agents is particularly advantageous, since these esters are readily obtained from the higher alcohols.<sup>737</sup>

Dimethyl sulfate reacts with thiourea in two stages:



When a mixture of 76 g. thiourea (1 mole), 63 g. dimethyl sulfate (0.5 mole) and 50 cc. of water is cautiously warmed a vigorous

reaction occurs. This is the first stage; the second is accomplished by boiling the mixture. The sulfate melts at 244° with decomposition and is useful for preparing methyl mercaptan and alkyl guanidines.<sup>22, 42, 1005, 1172</sup> The base can be set free by the addition of alkali. It melts at 79° to a clear liquid which decomposes into methyl mercaptan and cyanamide:



The base is seldom isolated as this and other reactions go on in solution.

The decomposition can be reversed. Methyl mercaptan is taken up almost quantitatively by a cold alcoholic solution of cyanamide. The mixture is allowed to warm up to complete the reaction and is then cooled to obtain the crystals.<sup>22</sup> Other examples of this will be given later when this is considered as a method of synthesis of S-alkylisothioureas. Thiourea is the starting point for the preparation of mercaptans by other procedures.<sup>86, 1383</sup>

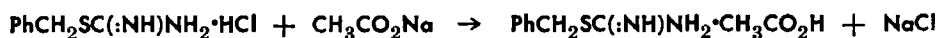
### *Identification of Alkyl Halides and Acids*

It has been proposed to use alkyl isothiuronium salts for the identification of alkyl halides. The halogen of the alkyl halide appears in the anion and can be identified by the methods of inorganic analysis. The salts have more or less characteristic melting points.<sup>120, 681</sup> Identification of the cation shows what alkyl was in the alkyl halide. It is convenient to convert the thiuronium salt to a picrate. The picrates are relatively insoluble and have rather high melting points. A mixture of 0.2 g. thiourea, an alkyl halide and 2 cc. of ethanol is heated for 2 minutes and picric acid added<sup>150</sup> or a solution of 1 g. thiourea and 1 g. of the alkyl halide in 100 cc. of ethanol is refluxed and picric acid added. Primary alkyl iodides and bromides react in a few minutes, secondary bromides and all chlorides, except specially active ones such as allyl, have to be heated several hours. The melting points of the picrates are high enough but are not as well scattered as could be desired. In addition to these picrolonates, 3,5-dinitrobenzoates,<sup>680, 681</sup> and styphnates<sup>681</sup> have also been prepared. The melting points of a number of these salts along with those of the picrates<sup>4, 820</sup> are given in the property list.

Another way to operate would be to use larger amounts of thiourea and alkyl halide and divide the S-alkyl isothiuronium salt solution into four or five parts. Picric acid may be added to one of these and other acids or their salts to the others. That would give four or five derivatives. It is unlikely that all of these would have the same melting points as those containing a different alkyl. A study of relative solubilities should give useful information.

Several heterocyclic halides can be identified using their isothiuronium salts.<sup>114, 1021, 1232</sup> Mono(chloromethyl)indigo has been identified by its reaction with trimethyl thiourea to form the isothiuronium derivative.<sup>1034</sup> The impurities in S-alkylisothiuronium salts can be detected by chromatography.<sup>573</sup>

Conversely acids may be identified by the melting points of their salts with a base of this type. The salts are obtained by double decomposition:



The reagent is readily prepared. A mixture of 126 g. benzyl chloride, 76 g. thiourea and 200 cc. of alcohol is heated under reflux for half an hour. The S-benzylisothiuronium chloride separates on cooling. It is filtered off and recrystallized from alcohol, or from a mixture of concentrated hydrochloric acid and an equal volume of water. It melts at 172–4° (sometimes at 146–8°). A 15% solution of this salt in hot alcohol is added to a concentrated solution of the sodium salt of the acid to be identified. Many of the salts of the organic acids are sparingly soluble and are precipitated in quite pure condition. They can be recrystallized from alcohol. Salts of a number of organic acids have been prepared and their melting points recorded.<sup>139, 244, 317, 572, 720, 797, 1303, 1321, 1322</sup> Starting with p-chlorobenzyl,<sup>284, 1261</sup> p-bromobenzyl<sup>293, 1211</sup> and p-nitrobenzyl<sup>1089</sup> halides the corresponding substituted S-benzylisothiuronium halides have been prepared and used for the identification of acids. The melting points of the salts of some aliphatic carboxylic acids are given in the property list.

The substitution of chlorine or bromine in the para position in the benzyl makes remarkably little difference. This method of identifying acids is attractive since the desired salts are readily prepared. As will be seen from the large number of salts cata-

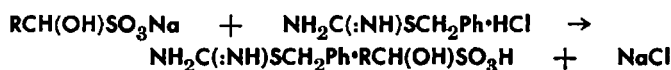
logged in the property list, it has been extensively used. A serious drawback is the fact that the compounds are salts and as such do not have sharp and reliable melting points. In cases where the melting points of the same salts have been reported by different observers there is sometimes serious disagreement. Then too, the spread of the melting points is less than could be desired.<sup>80, 124, 582, 696, 756, 835, 856, 1123, 1129, 1160, 1178, 1208, 1248</sup>

S-Benzylisothiuronium salts are particularly useful for the identification of sulfonic and sulfinic acids for which other methods are lacking. The melting points of the salts are more widely scattered. Data for a number are given in the property list.<sup>18, 52, 119, 175, 178, 192, 193, 212, 549, 650, 766, 937, 1085, 1259, 1333, 1336, 1384</sup>

Aldehydes and ketones combine with sodium bisulfite to form the sulfonic salts:



These salts undergo double decomposition with S-benzylisothiuronium chloride:



The resulting salts have satisfactory melting points and serve to identify the aldehydes and ketones.<sup>53, 232, 1335</sup> The melting points of some of these derivatives are in the property list.<sup>53, 232, 1335</sup>

Good results have been obtained with mono-alkyl sulfates  $\text{RSO}_3\text{H}$ . The data for some of these are given in the property list.<sup>47</sup> Since monoalkyl sulfates,  $\text{ROSO}_3\text{H}$ , can be obtained in great numbers by the addition of sulfuric acid to unsaturates, this method of identification is of considerable importance.

The three sulfobenzoic acids may be identified by their S-benzylisothiuronium salts.<sup>178, 1260, 1320</sup>

It is possible that the use of S-alkyl isothiuronium salts for identification can be extended to some inorganic acids. The application of the method is simple since the S-alkyl isothiuronium salt does not need to be isolated but can be used in the solution in which it is prepared. In fact the more soluble ones that would be difficult to isolate may be the most useful. Thiourea, water, and an excess of a volatile alkyl bromide are heated under reflux until the reaction is judged to be complete. The excess of the

alkyl halide is allowed to escape. The solution that remains is ready for use. Portions of it may be diluted to appropriate normalities. By adding to these a salt of the acid that is to be identified, beginning with the weakest, the approximate solubility of the new salt can be ascertained as well as its melting point.

S-benzylisothiuronium chloride has been recommended as a standard substance for test analyses for carbon, hydrogen, nitrogen, chlorine and sulfur since it is easily obtained pure.<sup>324</sup>

Thiourea reacts with polyethylene glycol bromohydrin to give thiuronium salts.<sup>808</sup> Tertiary alcohols may be identified as their S-alkylisothiuronium picrates after first converting them to the corresponding alkyl halide.<sup>1151</sup>

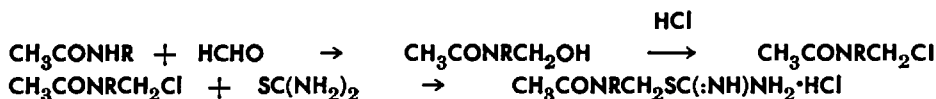
The S-benzylisothiuronium salts of several organic acids have been identified by X-ray powder diffraction patterns.<sup>923</sup> Some of the salts have been used to resolve racemic fatty acids.<sup>1002</sup>

The bromide<sup>613</sup> and picrate<sup>1029</sup> of the *i*-propyl base, *i*-PrSC(:NH)NH<sub>2</sub>, are said to be useful as drugs for raising blood pressure. This base can be made also by adding *i*-propyl mercaptan to cyanamide: <sup>613, 636, 1029</sup>



The higher mercaptans can be added to cyanamide to produce useful bases of this type.<sup>621</sup> The decyl and dodecyl salts, DecSC(:NH)NH<sub>2</sub>·HBr, m. 99° and DodSC(:NH)NH<sub>2</sub>·HBr, m. 111°, are said to have bactericidal and emulsifying properties.<sup>1029</sup> Other salts of these bases are claimed as wetting, dispersing and emulsifying agents.<sup>590a</sup> The α-, β- and γ-chloroalkyl cyanides and thiourea give the cyano-salts NC(CH<sub>2</sub>)<sub>n</sub>SC(:NH)NH<sub>2</sub>·HCl. The β- and γ-salts melt at 166° and 127°.<sup>907</sup>

A methylol amide, formed *in situ*, may be condensed with thiourea in the presence of an acid:<sup>617</sup>



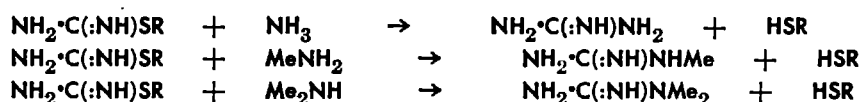
Chloroacetone reacts vigorously with thiourea and with 1,3-diphenylthiourea.<sup>992</sup> A halomethyl ester, RCO<sub>2</sub>CH<sub>2</sub>Cl, in which the acid contains more than 14 carbon atoms reacts with thiourea to give a wetting agent.<sup>1222b</sup> Neither ortho nor para nitrochlorobenzene reacts with thiourea, but 2,4-dinitrochlorobenzene and

picryl chloride give what appear to be the S-isothiuronium compounds.<sup>1275</sup>

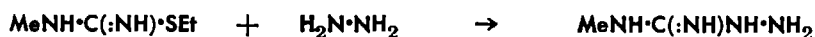
An alkyl mercury halide,  $\text{RHgX}$ , reacts with thiourea, or its derivatives, as if it were an alkyl halide. The products are said to be fungicides and bactericides.<sup>405</sup> The reaction of mercury halides with thiourea in melts has been investigated and the systems characterized.<sup>844</sup>

As was brought out in Vol. I, pages 32–5, the addition of an alkyl halide to thiourea is the initial step in a favorite method for preparing mercaptans. The addition of alkali to the salt frees the base which decomposes liberating the mercaptan. Even an amino mercaptan, such as  $\text{CH}_3\text{CH}(\text{SH})\text{CH}_2\text{NMe}_2$ , can be made in this way.<sup>1050</sup> For other examples, see under the preparation of mercaptans.

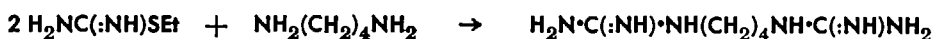
The S-alkylisothiureas are convenient starting materials for the preparation of substituted guanidines. The reaction of ammonia gives guanidine, an amine gives a substituted guanidine:



The alkyl that is joined to the sulfur is eliminated as a mercaptan and does not enter the guanidine. Hence the most commonly used are the S-methyl- 22, 139, 743, 1005, 1008, 1121, 1382 and S-ethyl- 130, 1254 isothiureas which are conveniently prepared from dimethyl sulfate or ethyl bromide. If hydrazine displaces the  $-\text{SR}$  an aminoguanidine results: <sup>718</sup>

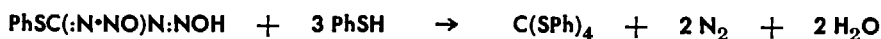


A diamine leads to a diguanide: <sup>1254</sup>



By starting with suitably substituted thioureas almost any desired substituted guanidine can be synthesized.<sup>957, 1121, 1122</sup>

The dinitroso- derivative of S-phenylisothiurea reacts with alkali mercaptide to give a tetrathioorthocarbonate: <sup>21</sup>



An alkylisothiurea can be oxidised directly to the sulfonic acid: <sup>12b</sup>



A convenient way to prepare alkyl sulfonyl chlorides is to pass chlorine into a cold water solution or suspension of S-alkylisothiurea salts: <sup>411, 673, 675, 1231</sup>



Oxidation by hydrogen peroxide in the presence of alkali gives a salt of the sulfonic acid.<sup>723c</sup>

### ACYL DERIVATIVES OF THIOUREA

An acyl derivative of thiourea,  $\text{RCONHCSNH}_2$ , is a thioureide of the acid  $\text{RCOOH}$ .

Mono-acetylthiourea is obtained by warming thiourea with acetanhydride. The melting point was given at first as  $11.5^\circ$  <sup>948a</sup> which was copied into books as  $115^\circ$ , but  $165^\circ$  is the correct figure.<sup>1024</sup> It is found in the mother liquor when thiourea is prepared from cyanamide by the action of thioacetic acid.<sup>68, 1024</sup>

When thiourea is boiled with two molecules of acetanhydride both mono- and di-acetyl derivatives are formed. The maximum yield of the mono- is 47% at 2.5 minutes while that of di- is 50% at 10 minutes. The yield of the diacetyl can be raised to 78% by the addition of hydrogen chloride or sulfuric acid. The structure of the monoacetyl is shown to be  $\text{CH}_3\text{CONH} \cdot \text{CS} \cdot \text{NH}_2$  by its decomposition into acetamide,  $\text{CH}_3\text{CONH}_2$ , and the decomposition products of thiocyanic acid, when it is heated to  $190^\circ$ . It is desulfurized to acetylurea.<sup>1374c</sup> When thiourea is acetylated under certain conditions a yellow compound is obtained, melting at  $150\text{--}5^\circ$ . This appears to be a molecular compound of the mono- and di-acetyl derivatives.<sup>587b, 752</sup> Diacetylthiourea dissolves in cold aqueous sodium hydroxide from which it is precipitated by acid unchanged. If the alkaline solution is heated before the acid is added, the precipitate is the monoacetyl derivative.<sup>587b</sup> Monoacetylthiourea melts at  $165^\circ$ , the diacetyl- at  $152^\circ$ .<sup>752</sup>

Heating an acid with half a molecule of thiourea is an excellent way to prepare an acid amide.<sup>268</sup>



When acetyl chloride is added to a saturated solution of thiourea in acetone a compound is precipitated which appears to be  $\text{MeCOS} \cdot \text{C}(:\text{NH})\text{NH}_2 \cdot \text{HCl}$ . It melts at  $109^\circ$  with effervescence

and is decomposed by water, regenerating thiourea. It gives a picrate melting at 120°. On heating it is changed to acetylthiourea with loss of hydrogen chloride.<sup>306</sup>



In pyridine solution thiourea and acetyl chloride give acetyl thiourea directly.<sup>308a</sup>

Often acylation occurs first on sulfur, when thiourea is treated with an acyl halide. After heating and sometimes on just merely standing the acyl group transfers from the sulfur to the nitrogen.<sup>306, 308a, 1145</sup> When an S-alkylpseudothiourea is heated with an acyl halide direct N-acylation occurs.<sup>202, 308b, 1148</sup> Diacyl derivatives of S-alkylisothiureas have been prepared in this way.<sup>202, 1148</sup> Refluxing thioureas with an acid in pyridine gives the acyl derivative<sup>412</sup> which can be made, the other way around, by the addition of ammonia to acetylisothiocyanate:<sup>904</sup>



The further action of ammonia gives acetamide.<sup>376</sup>

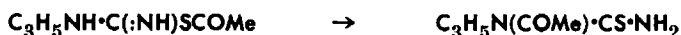
The potassium derivative of thiourea,  $\text{HN}\cdot\text{C}(\text{SK})\text{NH}_2$ , gives acetyl thiourea with acetanhydride.<sup>1374e</sup> This is more fully discussed in the chapter on thiocyanates.

A number of acyl derivatives of thiourea and of methylthiourea have been made by refluxing solutions of the acyl chlorides and thioureas in toluene. These compounds have some hypnotic action.<sup>916</sup>

Allylthiourea and acetyl chloride combine:



When the hydrochloric acid is taken out by alkali isomerization takes place:



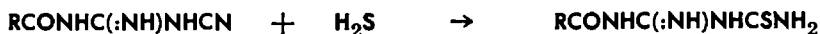
This melts at 96° and isomerizes further to  $\text{C}_3\text{H}_5\text{NH}\cdot\text{CS}\cdot\text{NH}\cdot\text{COMe}$  which melts at 74°.<sup>308a</sup>

Succinic anhydride and thiourea give the monothioureide,  $\text{HOOCCH}_2\text{CH}_2\text{CONH}\cdot\text{CS}\cdot\text{NH}_2$ , m. 211°. A similar compound melting at 223° is formed from citraconic acid.<sup>1007</sup> A mixture of phthalic anhydride and thiourea heated to 130° melts and then solidifies. The product is phthalic acid monothioureide, m. 172°,



which on further heating decomposes into phthalimide, carbon oxysulfide and ammonia.<sup>1009</sup> The double thiourea,  $\text{H}_2\text{NCS}\cdot\text{NH}\cdot\text{NH}\cdot\text{CS}\cdot\text{NH}_2$ , and its mono- and di-alkyl derivatives react with acetyl chloride; the products are cyclic.<sup>504</sup>

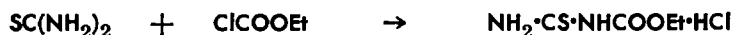
Acyl-guanyl-thioureas may be synthesized as follows: <sup>1228b</sup>



A number of acyl-substituted phenylthioureas of the formula  $\text{R}'\text{CONHCSNHR}$  where R is a hydroxycarboxyphenyl residue and R' is phenyl, tolyl, carbalkoxytoluyl or carbalkoxybenzoyl group, have been prepared.<sup>417</sup>

Thiourea reacts with two molecules of xanthidrol to form  $\text{SC}(\text{NHCH}(\text{C}_6\text{H}_4)_2\text{O}_2)_2$ , m.  $170^\circ$ .<sup>415</sup> Methylene camphorthiourea melts at  $213-4^\circ$ .<sup>78</sup>

Textile assistants are said to be obtained by reacting dichloromethyl ether with an amide and then with thiourea.<sup>1222c</sup> Thiourea and dibromobarbituric acid give  $\alpha$ -thiopseudouric acid.<sup>672, 1296</sup> Thiourea and ethyl chloroformate unite:



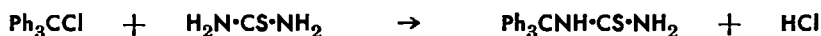
This melts at  $117^\circ$ .<sup>992</sup> According to a later study the reaction is somewhat complicated. The addition product of thiourea and ethyl chloroformate, apparently  $(\text{NH}_2)_2\text{CS}(\text{CO}_2\text{Et})\text{Cl}$ , reacts with sodium bicarbonate to form  $(\text{NH}_2)_2\text{CS}(\text{CO}_2\text{Et})\text{OCO}_2\text{H}$  which gives  $\text{NH}_2\cdot\text{CS}\cdot\text{N}(\text{CO}_2\text{Et})_2$ . This breaks down:



With alcoholic hydrochloric acid it is partially hydrolyzed: <sup>307</sup>



Triphenylmethyl chloride reacts with thiourea like an acid chloride: <sup>900</sup>



#### DESULFURIZATION

Oxidation may convert thiourea into urea.<sup>931</sup> Substituted thiourea may be desulfurized to the corresponding ureas by treatment with lead hydroxide <sup>504, 569b, 1138</sup> or mercuric oxide.<sup>895, 1138, 1370</sup>

Treated with mercuric, lead or silver oxide in alkaline solution

thiourea loses hydrogen sulfide; leaving cyanamide: <sup>465, 569b, 931, 1331</sup>

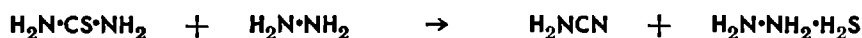


This reaction goes so well that an ammoniacal solution of silver nitrate may be used for titrating thiourea.<sup>1331</sup> It has been recommended as a preparation method for cyanamide.<sup>234, 1354</sup> Urea is also formed.<sup>1052a, 1138</sup> Treated with lead acetate in alkaline solution,  $\text{PhNHCSNH}_2$  is desulfurized to  $\text{PhNHCN}$ ; a similar reaction takes place with the p-chlorophenyl and p-tolyl derivatives.<sup>1194</sup> Freshly prepared mercuric oxide converts thiourea in alcoholic solution to cyanamide.<sup>68</sup> In presence of ammonia a part of this goes to guanidine and a part to dicyandiamide.<sup>1138</sup> As a qualitative test add ammonia and silver nitrate. A black precipitate indicates the presence of thiourea, a bright yellow one cyanamide.<sup>157</sup>

It has been suggested that the desulfurization is initiated by the addition of the metal hydroxide, or salt, to the sulfur atom by coordinate bonds. Water and metal sulfide are split off leaving carbodi-imide which rearranges to cyanamide or takes up water to form urea.<sup>1098</sup>

In the oxidation of  $>\text{CS}$  to  $>\text{CO}$  in nitrogen compounds by hydrogen peroxide the presence of alkali is important, and the speed depends on the concentration of the alkali.<sup>723a</sup>

Diethylthiourea,  $(\text{EtNH})_2\text{CS}$ , is desulfurized very slowly by mercuric oxide, not at all by lead oxide. Diethylurea is formed. In the presence of ethylamine, triethylguanidine is obtained.<sup>569b</sup> 1,3-Dibenzylthiourea<sup>1255</sup> and ethylenethiourea<sup>740</sup> are desulfurized to the corresponding ureas by mercuric oxide. Symmetrical disubstituted thioureas,  $\text{RNH}\cdot\text{CS}\cdot\text{NHR}$ , are desulfurized to the corresponding ureas provided one, or both of the groups, is aromatic. Aliphatic thioureas and aromatic of the types,  $\text{R}_2\text{N}\cdot\text{CS}\cdot\text{NH}_2$ ,  $\text{R}_2\text{N}\cdot\text{CS}\cdot\text{NHR}$  and  $\text{R}_2\text{N}\cdot\text{CS}\cdot\text{NR}_2$  are not so desulfurized.<sup>184, 305b</sup> A mono-alkylthiourea goes to a trialkyl melamine when desulfurized with lead or mercury oxide.<sup>569d</sup> Hydrazine removes hydrogen sulfide from thiourea: <sup>259</sup>



Thiourea is decomposed completely by Raney nickel. The products are ammonia, methyl amine, hydrogen sulfide and a

little methane. Benzylthiourea gives hydrogen sulfide, ammonia, methyl amine and toluene:<sup>123</sup>

Heated in glycerol solution, thiourea decomposes into ammonia, ammonium sulfide, cyanide and thiocyanate, and persulfocyanic acid.<sup>236</sup> In aqueous solution at room temperature, hydrogen sulfide is split off leaving cyanamide. This is the reversal of one method of formation.<sup>1138</sup>

### OXIDATION

Quite different products are obtained with different reagents or with the same reagent under different conditions.

Ozone gives sulfur and sulfuric acid with thiourea;<sup>1378</sup> hydrogen peroxide may do the same.<sup>542</sup> Benzoyl peroxide causes the separation of sulfur.<sup>1315</sup>

Electrolytic oxidation in concentrated hydrochloric acid gives the salt,  $\text{NH}_2(\text{NH}:)\text{C}\cdot\text{S}_2\cdot\text{C}(:\text{NH})\text{NH}_2\cdot 2\text{HCl}$ , from which the thiourea can be regenerated by reduction.<sup>397, 398</sup> In nitric acid the corresponding dinitrate is obtained. Phenylthiourea gives more complicated results.<sup>884</sup>

$[\text{2,4-(O}_2\text{N)}_2\text{C}_6\text{H}_3]_2\text{S}_2$  is formed from thiourea and 2,4-dinitro phenyl chloride by oxidation of the mercaptan first produced. The disulfide is produced by strong oxidising agents not only in the presence of strong acids but by iodine even in neutral solution.<sup>1251, 1374d</sup> The estimation of thiourea by oxidimetric titration has been considered under analysis. More complete oxidation gives urea and sulfuric acid.<sup>1374d</sup>

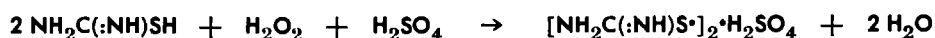
Thiourea is decomposed by sodium hypochlorite or hypobromite but not as readily as is urea.<sup>235, 243, 1052a</sup> Nitric and nitrous acids and ethyl nitrate and nitrite react with thiourea in rather complicated ways.<sup>222a, 223, 230</sup> The reaction is in part: <sup>305a, 1374d</sup>



Under proper conditions thiourea may be used for the gasometric estimation of nitrous acid.<sup>230</sup> This acid converts 1,1-phenylmethylthiourea into a cyclic compound.<sup>1098</sup>

Unless special precautions are taken, hydrogen peroxide breaks up thiourea into ammonia, carbon dioxide, sulfur and sulfuric acid. From allylthiourea some allyl amine and formic acid are produced.<sup>542</sup> If thiourea and an equivalent of acid are dissolved

in 50% alcohol and the calculated amount of hydrogen peroxide is added the salt of the disulfide is formed: <sup>112</sup>

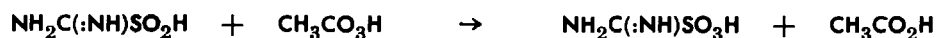


The free base is unstable. <sup>112</sup>

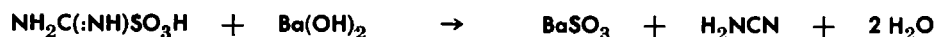
When powdered thiourea is added slowly to 6% hydrogen peroxide at 0° crystals of aminoiminomethane sulfinic acid,  $\text{NH}_2\text{C}(\text{:NH})\text{SO}_2\text{H}$ , separate. <sup>64</sup> One may use 30% peroxide, filter off and dry the crystals at 25–30°; <sup>1315</sup> or hydrogen peroxide may be fed into a saturated aqueous solution of thiourea until the latter is consumed. Then more thiourea and peroxide are added in succession, keeping the temperature below 10°. The product crystallizes out and is filtered off and dried. <sup>539</sup> Formamidine sulfinic acid, as it is also called, is moderately soluble in water at 30° but only slightly so at 0°. <sup>1346</sup> It melts, or rather decomposes, at 125–40° according to the rate of heating. The pure solid may be stored for a year in a cool dry place. A dilute water solution shows 40% decomposition at room temperature in eight days. Although it is spoken of as an acid its aqueous solution is neutral to litmus and Congo red. <sup>112</sup> It is sometimes called thiourea dioxide and its formula written,  $(\text{NH}_2)_2\text{CSO}_2$ . <sup>112</sup>

The remarkable thing about this compound is that, although it is itself an oxidation product, it is a powerful reducing agent. <sup>112</sup> In alkaline solution it precipitates cadmium, lead, tin, antimony, copper, silver, mercury, bismuth and arsenic from solutions of their salts. <sup>112</sup>

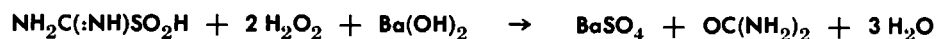
Formamidine sulfinic acid is oxidised by peracetic acid to formamidine sulfonic acid: <sup>112</sup>



This acid may be prepared directly by oxidising thiourea with excess of peracetic acid. It is split by barium hydroxide into barium sulfite and cyanamide:



Formamidine sulfinic acid, barium hydroxide and hydrogen peroxide give barium sulfate and urea: <sup>112</sup>



A substituted thiourea is oxidised similarly by hydrogen per-

oxide to the corresponding formamidine sulfinic acid.<sup>723b</sup> Thiourea can be photo-oxidised in the presence of protoporphyrin to give formamidine sulfinic acid; allyl thiourea can be oxidised in the same manner.<sup>1120</sup>

Thiourea and 1,3-diethyl thiourea are used as reducing agents in the reduction-oxidation system described for the bulk polymerization of unsaturated compounds like styrene.<sup>709</sup>

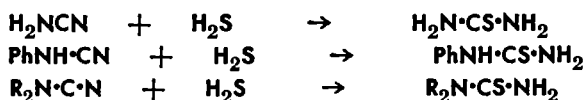
### Substituted Thioureas

These have been prepared in considerable numbers and varieties by several methods. A comprehensive review of the methods of preparation and physiological properties of substituted thiourea has been written by Dorothy C. Schroeder.<sup>1152</sup>

#### PREPARATION

##### *From Cyanamides and Mercaptans*

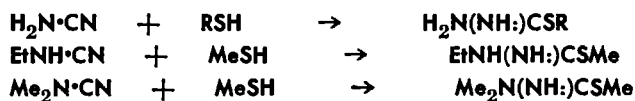
Cyanamide reacts with hydrogen sulfide, so do its substitution products: <sup>1101, 1347</sup>



The reversal of this reaction is accomplished by heating a thiourea with moist lead oxide: <sup>154</sup>



A mercaptan adds to cyanamide and to its derivatives: <sup>1037c, 1125, 1126</sup>



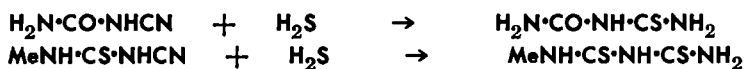
The addition of a mercaptan to cyanamide is the reversal of the widely used preparation of mercaptans by the decomposition of S-alkylisothiureas:



The S-alkylisothiurea can be made by the reaction of an alkyl halide on the sodium derivative of thiourea: <sup>518</sup>



The addition of hydrogen sulfide to cyanurea and to methylthiocarbaminecyanamide gives monothiobiuret and methylthiobiuret: <sup>541</sup>



The addition of hydrogen sulfide to dicyanamide forms guanylthiourea: <sup>1037c</sup>



Dithiobiuret is formed when this reaction is conducted under pressure. <sup>1228a</sup>

An odd reaction which results in the formation of diphenylthiourea is the action of sulfur on N-methylenedianiline: <sup>154</sup>



Diphenylselenourea is made by the addition of hydrogen selenide to diphenylcarbodiimide: <sup>1421</sup>



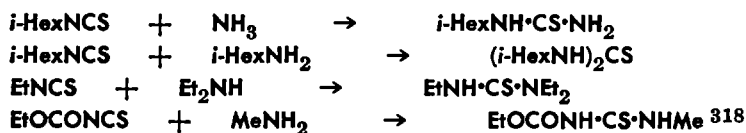
This holds for other carbodiimides.

### From Isothiocyanates

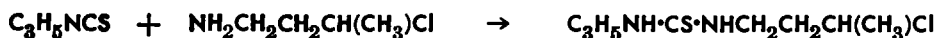
A simple way to prepare a substituted thiourea is the addition of ammonia or an amine to an isothiocyanate. An early example is the formation of thiosinamine from mustard oil:



This and many other examples will be taken up in Volume V. Mono, <sup>596b, 660, 687, 731, 877, 1105, 1107, 1199</sup> di <sup>171, 344, 687, 1105</sup> and tri-substituted thioureas are produced according to whether ammonia, a mono- or dialkyl amine <sup>762, 803</sup> is the addendum:

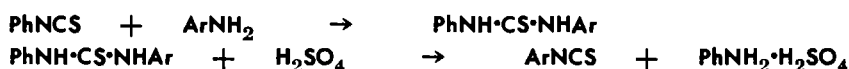


Since this is such a simple reaction and since most thioureas are solids, it has been used in the characterization of amines <sup>141, 150, 182, 432, 978, 1095, 1262, 1299</sup> and isothiocyanates. <sup>734</sup>



As this product is a halide and a thiourea there is a self-condensation resulting in a cyclic compound.<sup>838</sup> When the alkyl of the isothiocyanate contains 8 or more carbon atoms and the amine contains a water-solubilizing polar group the product is surface active.<sup>1108</sup>

The addition of an amine to a mustard oil may be reversed by boiling the thiourea with strong aqueous sulfuric acid:



Actually the hydrolysis product is a mixture of the two isothiocyanates. However, there is a partial exchange of function between the amine and the mustard oil.<sup>201</sup>

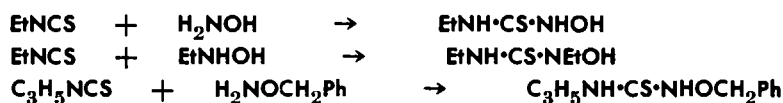
Benzoyl isothiocyanate unites readily with primary or secondary amines to give benzoyl derivatives of thioureas: <sup>319, 418, 941</sup>



Aryl amines give better results than alkyl. The benzoyl group is readily hydrolyzed off from the primary amine derivatives. Other RNHCSNHR compounds have been synthesized.<sup>1185</sup>

The effect of nuclear substituents on the reactivity of aryl isothiocyanates has been studied.<sup>153</sup>

Hydroxylamine and its alkyl derivatives unite with mustard oils to give hydroxythioureas: <sup>735, 736</sup>



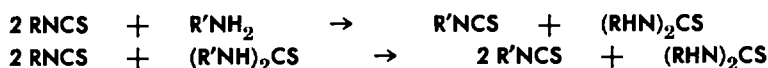
This will be discussed more fully under thiocyanates.

Diacetone amine unites with allyl mustard oil to give diacetone allylthiourea, m. 138°, <sup>1289</sup> and with ethyl isothiocyanate to form the corresponding ethyl derivative, m. 198.5°. <sup>683</sup>

A mustard oil combines with guanidine to form a guanylthiourea: <sup>433b, 1059, 1190, 1202</sup>



A high molecular weight sulfonic acid is obtained by adding an aminosulfonic acid to an isothiocyanate such as octadecyl. <sup>623</sup> Urethan formation and the following exchange reactions are the chief complications which occur in this reaction: <sup>1420</sup>



### *From Amines and Carbon Disulfide*

An important method of preparing symmetrical disubstituted thioureas starts with a primary amine and carbon disulfide. The first reaction is the formation of the dithiocarbamate salt:



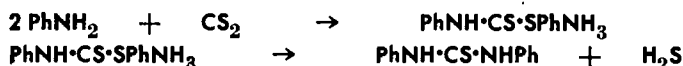
This salt loses hydrogen sulfide on heating:



The first reaction takes place spontaneously at room temperature; the second usually requires several hours heating on the water bath.<sup>569a, 753, 810, 1101, 1368</sup> The addition of some sulfur accelerates the reaction.<sup>478, 587a, 589, 1033</sup> Hydrogen peroxide is also a good catalyst for the reaction, since free sulfur is formed as a by-product instead of hydrogen sulfide.<sup>132a, 133</sup> Diarylthioureas have been made with the addition of iodine to the amine and carbon disulfide in pyridine.<sup>434, 435, 1033</sup> There has been much discussion of the mechanism of this reaction.<sup>140, 237, 249, 324b, 818</sup>

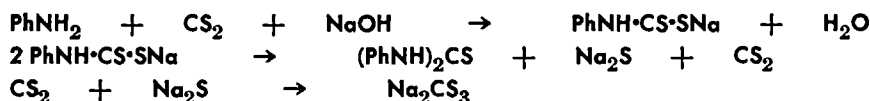
This method has been used for the lower alkyl amines,<sup>194, 421, 541, 569a, 660, 871, 1022, 1105, 1199, 1374g</sup> hexahydroanthranilic acid,<sup>356</sup> bornylamine,<sup>413, 1348, 1420</sup> and higher amines such as pentadecyl,<sup>666</sup> cetyl and oleyl.<sup>1339</sup> Cyclic thioureas are obtained from the ethylene diamine<sup>6, 489e, 740, 950, 1086</sup> and 2,3-butylene diamine<sup>1416</sup> but when the amino groups are separated by a longer chain the products are plastic polymers.<sup>247</sup> Tri- and tetramethyl-4-aminopiperidines yield thioureas.<sup>531</sup> The product from tetraamino-neopentane appears to be  $\text{C}[(\text{CH}_2\text{NH})_2\text{CS}]_2$ .<sup>8</sup> A mixture of mono- and di-ethyl amines and carbon disulfide gave triethylthiourea,  $\text{Et}_2\text{N}\cdot\text{CS}\cdot\text{NH}\text{Et}$ , m.  $26^\circ$  b.  $205^\circ$ .<sup>492</sup> From carbon disulfide, ammonium carbonate and alcohol, heated at  $160^\circ$  in a sealed tube, diethylthiourea  $(\text{EtNH})_2\text{CS}$ , m.  $77^\circ$ , is formed.<sup>647</sup> Acetone, carbon disulfide and ammonium hydroxide, standing in a closed vessel for three weeks at room temperature, combine to form pinacolylthiourea, m.  $240-3^\circ$ .<sup>543, 544</sup>

Diarylthioureas are particularly easy to prepare from the amine and carbon disulfide: <sup>441, 569a, 753, 831, 1037a</sup>





Dibenzylthiourea,  $(\text{PhCH}_2\text{NH})_2\text{CS}$ , m.  $114^\circ$ ,<sup>1255</sup> and *p,p'*-diphenoxyphenylthiourea,  $(\text{PhOC}_6\text{H}_4\text{NH})_2\text{CS}$ , m.  $172^\circ$ ,<sup>791</sup> have been prepared.  $\alpha$ -Methylbenzyl amine gives a mixture of the *dl*- and *meso*-forms of the dimethyldibenzylthiourea. The active forms can be made from the active amines. These melt at  $195.5^\circ$  and have the rotations,  $[\alpha]_D$   $22.5^\circ$  and  $-22.1^\circ$ .<sup>834</sup> A disulfonated thiourea can be obtained from an aminonaphthol sulfonic acid.<sup>649</sup> The addition of a small amount of ethyl potassium xanthate to the mixture improves the yield and quality of the thiourea.<sup>502, 503, 870</sup> A considerable excess of the aryl amine is said to be desirable.<sup>1186, 1188</sup> The reaction may be effected at the boiling point of the carbon disulfide<sup>525, 526</sup> or above that temperature.<sup>708</sup> The reaction time can be shortened and the yield increased by the addition of caustic alkali.<sup>85, 407, 1033, 1186, 1189, 1210, 1368</sup> The reactions may be formulated thus:



A convenient laboratory method is as follows: To a mixture of 372 g. aniline (4 moles) and 380 g. carbon disulfide (5 moles) add 400 cc. of 40% sodium hydroxide solution, with vigorous agitation and cooling, add 500 cc. of water to make the mixture more fluid, and filter off the diphenylthiourea,  $(\text{PhNH})_2\text{CS}$ . The yield is 534 g., 94%.<sup>1210</sup>

In certain cases alcohol may be used as a solvent for the starting materials. Alcohol is also recommended as a solvent if it is necessary to raise the reaction temperature.<sup>569b</sup>

Aniline refluxed with a suspension of a heavy metal trithiocarbonate: <sup>324a</sup>

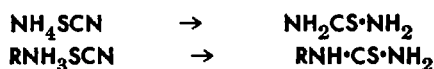


In cases where hydrogen sulfide is not readily separated from the dithiocarbamate the addition of hydrogen peroxide is advised. Thus 6.4 g. benzyl amine, 2.3 g. carbon disulfide and 17 cc. of 3% hydrogen peroxide give 5 g. of dibenzylthiourea.<sup>132b</sup> When aniline or  $\alpha$ -naphthylamine is melted with thiourea at  $150\text{--}80^\circ$ , the product is primarily the monosubstituted thiourea.<sup>967</sup> Rhodanturates react with sulfonated aromatic amines to give sulfonated thioureas.<sup>1212</sup> The  $\alpha$ -amino-acids resulting from the hydrolysis of a protein may be converted into thioureas.<sup>595</sup>

A trisubstituted thiourea is obtained by splitting off hydrogen sulfide from the dialkylamine salt of a dithiocarbamic acid derived from a monoalkyl amine.<sup>612, 633</sup>

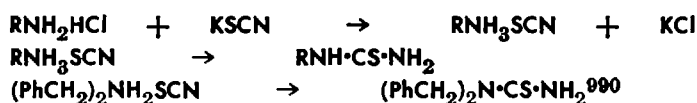
*By Isomerization. of Substituted Ammonium Thiocyanates*

As ammonium thiocyanate isomerizes to thiourea, so substituted ammonium thiocyanates go to the corresponding thioureas:



When aniline is added to an ether solution of thiocyanic acid aniline thiocyanate separates out and isomerizes to phenylthiourea. The equilibrium seems to be more favorable than with the unsubstituted.<sup>764</sup> It is not necessary to prepare the free thiocyanic acid. Mono- and unsymmetrical di-substituted thioureas are obtained by heating an amine or an amine hydrochloride with ammonium thiocyanate.<sup>626, 837a, 1037c, 1355</sup>

Some of the more complex thioureas which have been prepared by this method include: 8-(5,6-dimethyl quinolyl)-phenylthiourea,<sup>360</sup> (3-hydroxy-4-carbomethoxy phenyl) thiourea, and sulfanilamide-N-4-thiourea derivatives.<sup>811, 1410</sup> Aniline hydrochloride and ammonium thiocyanate are heated in aqueous solution. The aniline thiocyanate is converted to phenylthiourea which settles out at 100° as an oily layer.<sup>738</sup> The presence of an inert solvent is said to be advantageous.<sup>611, 642</sup> As the function of the ammonium thiocyanate is to furnish the thiocyanate ion, an alkali thiocyanate will serve as well: <sup>620, 950, 990</sup>



Instead of preparing the amine hydrochloride beforehand it is more convenient to mix one mole of the amine with 1.3 of hydrochloric acid and 1.1 of potassium thiocyanate and heat. Some 60 thioureas were prepared in this way.<sup>277</sup>

It has been found that dilute sulfuric acid accelerates the isomerization of methylamine thiocyanate, but does not effect the rearrangement of benzylamine thiocyanate.<sup>508</sup>

As in the case of the transformation of ammonium thiocyanate into thiourea, the isomerization of an alkylammonium thiocyanate into an alkylthiourea is a slow reaction tending toward

an equilibrium. The results of a quantitative study are in Table 3.4.

TABLE 3.1  
*Percentages of Alkylthioureas Formed by Heating  
Alkylammonium Thiocyanates*

|                  | MeNH <sub>2</sub> | EtNH <sub>2</sub> | Me <sub>2</sub> NH | Et <sub>2</sub> NH |
|------------------|-------------------|-------------------|--------------------|--------------------|
| At 130° 5¾ hours | 32.9%             | 29.4%             | 16.9%              | 3.6%               |
| 130° 8 hours     | 38.5              | 34.4              | 16.7               | 3.9                |
| 150° 5¾ hours    | 49.4              | 48.7              | 14.4               | 6.6                |

The reaction is much slower with the dialkyl amines.<sup>1105</sup>

When thiourea is heated with an aryl amine there is partial replacement of ammonia and formation of some monoarylthiourea.<sup>1083</sup> This goes further with certain substituted thioureas:<sup>942</sup>



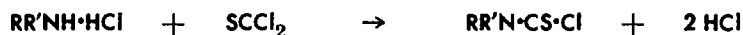
The equilibria in the thermal isomerization of Me<sub>2</sub>NH<sub>2</sub>SCN has been investigated in detail at various temperatures and under various conditions.<sup>1196</sup>

#### *From Thiophosgene*

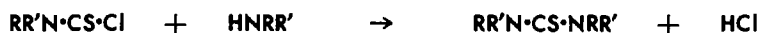
Tetra N-substituted thioureas are prepared by treating dialkyl amines with thiophosgene:



This reaction may go in two stages, particularly if the amine hydrochloride is the starting material:



In neutral or alkaline solution a further reaction takes place:<sup>71a, 96, 98, 281, 343, 344, 597, 608, 644, 924</sup>



Methylethylamine is converted into N,N'-dimethyl-N,N'-diethyl thiourea, MeEtN·CS·NEtMe, b<sub>12</sub> 124–5°.<sup>804</sup>

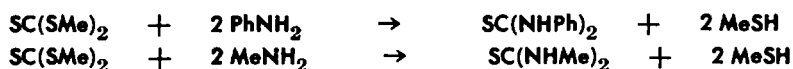
The thiophosgene method is usually used where other methods fail, such as in the case of aromatic amines with strong negative

substituents or in the conversion of secondary amines to the corresponding symmetrical thioureas.<sup>342, 503</sup>

An aromatic nitroso-derivative is converted to a diarylthiourea by sodium trithiocarbonate:<sup>1187</sup>



Trithiocarbonates of metals, of which the sulfides are insoluble, are acted upon by aromatic amines.<sup>12c, 14, 1234a</sup> Alkyl trithiocarbonates react with amines to give thioureas and mercaptans:<sup>282</sup>



Sulfides may react with a double bond. Cyanothiurea has been prepared by heating a metal dicyanamide with hydrogen sulfide.<sup>873</sup> The sulfur of a 1,3-disubstituted thiourea adds to an acetylenic bond to give a pseudothiurea salt in acid medium.<sup>190</sup>

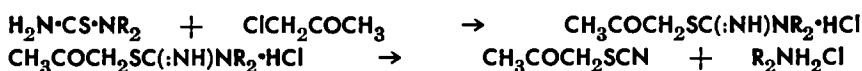
#### REACTIONS OF SUBSTITUTED THIOUREAS

Alkyl halides add to substituted thioureas as to the parent compound. The products are salts from which the free bases can be liberated by alkali.<sup>265</sup>

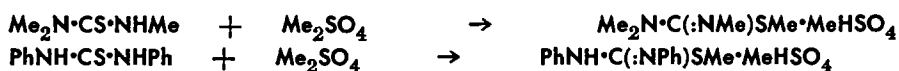
Some of the more complex thiourea derivatives prepared by this method include: thiourea derivatives of substituted glucose,<sup>506</sup> several heterocyclic thioureas, several substituted phenyl thioureas,<sup>214b, 515, 1356, 1415</sup> and thiobiguanides.<sup>2, 506, 903</sup> Thiosinamine takes up ethyl iodide.<sup>1370</sup> The product  $\text{H}_2\text{C}:\text{CHCH}_2\text{NH}\cdot\text{C}(\text{:NH})\text{SEt}\cdot\text{HI}$ , m.  $72^\circ$ ,<sup>887a</sup> called Tiodine has been proposed as a therapeutic agent.<sup>321, 742</sup> Phenyl- and 1,3-diaryl-thioureas combine with methyl and ethyl iodides.<sup>88, 1047, 1387, 1388</sup> Polymeric compounds result when a dihalide reacts with a double thiourea.<sup>591</sup> The DL- and meso- forms of 1,3-di- $\alpha$ -methylbenzylthiourea,  $(\text{PhMeCHNH})_2\text{CS}$ , take up methyl iodide slowly but quantitatively.<sup>970</sup> Triethylthiourea takes up ethyl iodide. The free base,  $\text{Et}_2\text{N}\cdot\text{C}(\text{:NEt})\text{SEt}$ , is unusually stable<sup>492</sup> since the absence of hydrogen on the nitrogen precludes the splitting off of a mercaptan. Tetramethyl-, tetraethyl- and tetrapropyl bases have been prepared.<sup>281</sup> The addition of methyl iodide to  $\text{SC}(\text{NMe}_2)_2$  and to  $\text{MeSC}(\text{:NMe})\text{NMe}_2$  leads to identical iodides. The same iodide is obtained by adding ethyl iodide to  $\text{MeS}\cdot\text{C}(\text{:NEt})\text{NMe}_2$  and methyl iodide to  $\text{MeS}\cdot\text{C}(\text{:NMe})\text{NEt}_2$ .  $\text{Me}_2\text{N}(\text{MeS})\text{C}:\text{NEt}_2\text{I}$

and  $\text{Et}_2\text{N}(\text{MeS})\text{C}:\text{NMe}_2\text{I}$  are isomeric. They are both quaternary ammonium salts but differ in structure.<sup>805</sup>

The reaction of chloracetone or bromacetophenone with an unsymmetrical disubstituted thiourea may be supposed to proceed regularly but the thiuronium salt, if formed, is unstable and breaks up into a thiocyanate and a salt of the disubstituted amine: <sup>1229</sup>



Dimethyl sulfate can be used in place of an alkyl iodide: <sup>278, 801, 802, 803</sup>

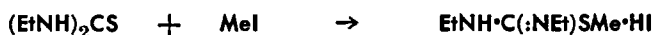


Monothiobiuret combines with methyl iodide: <sup>784</sup>

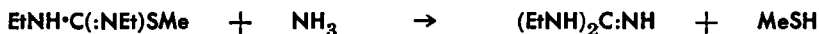


A dithiobiuret reacts with benzyl chloride.<sup>431</sup> The nitrosothiourea,  $\text{MeNH}\cdot\text{CS}\cdot\text{N}(\text{OH})\text{Me}$ , gives an unstable addition compound,  $\text{MeN}:\text{C}(\text{SEt})\text{N}(\text{OH})\text{Me}\cdot\text{HI}$ , with ethyl iodide.<sup>786</sup> Thiobiurets can be cyclized to substituted 1,2,4-thiadiazoles.<sup>780c</sup> S-Alkylisothiourea reacts with alpha and gamma bromoacetates to give substituted pyrimidines.<sup>310</sup> Mercaptoacetic acid reacts with S-alkyl, -aryl, or arylalkylisothioureas to give thiazolidones. The mechanism of the reaction is believed to involve addition of the acid to isothiourea, elimination of  $\text{RSH}$ , and cyclization by elimination of water.<sup>13</sup>

A symmetrical dialkylthiourea reacts readily with an alkyl halide:



With ammonia this gives methyl mercaptan and diethyl guanidine: <sup>957</sup>



The mercaptan is the same that is obtained from the addition of the same halide to thiourea. Hence for preparing mercaptans, the cheaper thiourea is used but this offers a way of transforming N,N'-substituted thioureas into N,N'-disubstituted guanidines. If, instead of ammonia, an alkyl amine is reacted with the

N,N'-dialkylthiourea, the product is the N,N',N''-trisubstituted guanidine.<sup>801, 802, 803, 942</sup> With trimethylene bromide a double thiourea,  $\text{EtNH}\cdot\text{C}(\text{:NEt})\text{SCH}_2\text{CH}_2\text{CH}_2\text{S}\cdot\text{C}(\text{:NEt})\text{NHEt}$ , is obtained but with ethylene bromide the second bromine atom may react with a hydrogen on one of the nitrogens giving a cyclic compound.<sup>88, 275, 957, 1387, 1388</sup>

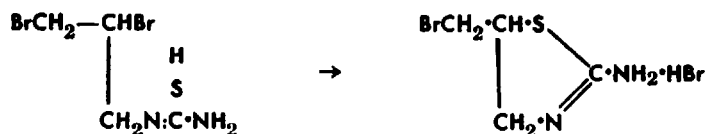
The rates of addition of methyl and ethyl iodides to substituted thioureas have already been given in the section on the reactions of thiourea.<sup>472</sup>

Thiosinamine forms a deliquescent hydrochloride with methyl chloroformate.<sup>305d</sup> It reacts also with acetylchloride:



With weak alkali this is transformed into  $\text{C}_3\text{H}_5\text{N}(\text{COCH}_3)\text{CS}\cdot\text{NH}_2$ , m.  $96^\circ$ . When this is melted, it becomes  $\text{C}_3\text{H}_5\text{NH}\cdot\text{CS}\cdot\text{NHCOCH}_3$ , m.  $74^\circ$ .<sup>308a</sup>

Thiosinamine takes up two atoms of iodine, two of bromine or a molecule of cyanogen.<sup>867a</sup> In the iodine compound one iodine atom is differently situated from the other since it is replaced by chlorine or by the cyano group by contact with silver chloride or cyanide.<sup>439b, 867a</sup> The same is true of one bromine atom of the dibromide. From the dibromide potassium hydroxide sets free an oil which is strongly basic and combines with hydrobromic acid to regenerate the dibromide.<sup>305d</sup> It appears that the 2,3-dibromopropylthiourea, first formed, has reacted with itself:



The addition of bromine to methyl-allyl-thiourea,<sup>305d</sup> to diallylthiourea,<sup>1139</sup> and to d-butylallylthiourea leads to analogous cyclic compounds.<sup>1306</sup> The pharmacological actions of the compounds of this group have been investigated.<sup>315, 1139, 1323</sup>

Thiosinamine unites with silicon tetrabromide to form  $\text{SiBr}_4\cdot 8(\text{C}_3\text{H}_5\text{NH}\cdot\text{CS}\cdot\text{NH}_2)$  <sup>1052d</sup> and with cyanogen to give a dicyanide which decomposes without melting at  $193^\circ$ .<sup>1088</sup> The union with iodine is so definite that thiosinamine may be titrated with iodine in acid solution.<sup>927</sup> It is oxidised by hydrogen peroxide to allylamine, ammonia, sulfuric and formic acids.<sup>542</sup> Phenylthiosinamine adds bromine, iodine and cyanogen.<sup>867a</sup>

Ethylpseudothiurea forms addition products with ethyl oxalacetate and with ethyl acetylcyanacetate.<sup>1380</sup> S-Alkylcyanopseudothiureas have been prepared by treating S-alkylpseudothiurea, suspended in acetone, with sodium hydroxide and cyanogen chloride.<sup>685</sup> The nitration of 2-alkylisothiuronium sulfate and nitrate salts yields the corresponding 2-alkyl-1- (or 3)-nitro-2-thiopseudourea.<sup>404</sup>

In dry pyridine  $\text{Me}_2\text{NCSNHMe}$  reacts with  $\text{HgO}$  to give a complex salt where four thioureas are apparently linked to mercury by their sulfur atoms. The reactions of thiourea derivatives in other solvents give different products; thus in ethanol the corresponding urea is produced, and in benzene an alkyl carbodiimide is formed.<sup>809</sup>

Symmetrical disubstituted thioureas in general lose hydrogen sulfide when their benzene solution is boiled with lead oxide or shaken with mercuric oxide: 794, 809, 1368



These carbodiimides are unstable liquids. Those with long alkyls, particularly secondary ones, are more stable.<sup>589, 1032, 1079, 1133, 1134, 1420</sup> An attempt to make the unsubstituted carbodiimide,  $\text{NH}:\text{C}:\text{NH}$ , by this method failed.<sup>931</sup> Heating diarylthioureas above their melting points is sufficient to drive off the hydrogen sulfide leaving the diaryl carbodiimides as polymeric resins. The desulfurization of biphenylenethiourea,  $-\text{C}_6\text{H}_4\cdot\text{C}_6\text{H}_4\cdot\text{NH}\cdot\text{CS}\cdot\text{NH}-$ , gives a black brittle resin.<sup>555</sup> Diphenylcarbodiimide is formed when thiocarbanilide reacts with diphenyldichloromethane: 213a

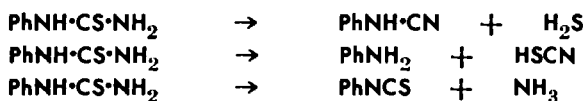


S-Ethyldiphenylisothiurea breaks up by dry distillation into mercaptan and diphenylcarbodiimide: 1387



S-Methyldi- $\alpha$ -naphthylisothiurea reacts similarly.<sup>377</sup>

Hydrolytic agents decompose phenylthiourea in three different ways:



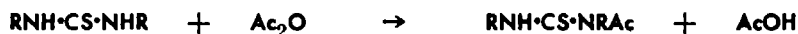
Acidity and other conditions determine which of these will predominate.<sup>201, 785</sup> The last reaction is a reversal of a common synthesis of thioureas.

Intramolecular rearrangements of certain substituted phenylbenzimidothioureas may take place.<sup>1064</sup> Phenylthioureas containing para-substituted tertiary amino groups may be synthesized by boiling the corresponding benzoyl imide with sodium hydroxide.<sup>1356</sup> Compounds of the type, 1,3-diaryl-2,4-dithioxo- $\delta$ -octahydroquinazoline, have been prepared by condensing N,N'-diaryl derivatives of thiourea with cyclohexanone.<sup>1147</sup>

Boiling thiocarbanilide with acetanhydride for 5–15 minutes gives a high yield of phenyl isothiocyanate: <sup>587a, 594, 1374b</sup>



Longer heating diminishes the yield. Below 80° acetylation takes place.<sup>587a</sup>



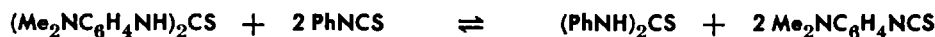
Heating two molecules of an acid with a substituted thiourea gives the N-substituted amide:



At 130–70° the yields are nearly quantitative; <sup>268</sup> at 270° they run from 60 to 80%.<sup>271</sup>

Formaldehyde condenses with disubstituted thioureas such as ethylenethiourea, dimethylethylene thiourea, and trimethylene-thiourea to give various straight chain and ring compounds.<sup>1237b</sup> High yields of nitriles are obtained when alkyl- or aryl-thioureas are heated with iron or copper in paraffin oil to 280°. <sup>304b</sup> Aryl-thioureas with  $\text{ArSO}_2\text{Cl}$  in pyridine give aryl cyanamides and sulfur.<sup>780a</sup>

An exchange may take place between a substituted thiourea and an isothiocyanate:<sup>1420</sup>



If thiocarbanilide is treated with methyl magnesium iodide and then with ethyl chlorocarbonate the product is the ester  $\text{SC}(\text{NPh-CH}_2\text{CO}_2\text{Et})_2$ , m. 146°, which can be hydrolyzed to the acid  $\text{SC}(\text{NPhCH}_2\text{CO}_2\text{H})_2$ , m. 144°. <sup>940</sup>

Nitrous acid gives different products with thioureas according to the conditions.<sup>785</sup> Ethylthiourea is partially converted to



ethylisothiocyanate. The same is true of the allyl.<sup>305a</sup> Aryl thioureas heated in chlorobenzene at 150° give good yields of 1,2,3, and 4 ringed aryl isothiocyanates.<sup>70</sup>

Ethylenethiourea is oxidised by iodine to dihydroglyoxaline disulfide periodide. Boiling this with water eliminates iodine and sulfur leaving the monosulfide.<sup>673.5</sup> In acid solution allylthiourea is oxidised by hydrogen peroxide to the disulfide, but in neutral solution to allylamine, ammonia, formic and sulfuric acids.<sup>542</sup> Thiazoles and thiadiazole are produced on oxidising certain thioureas with hydrogen peroxide or iodine.<sup>780a, 859</sup> The oxidation products of thiourea with bromine have been investigated.<sup>1097, 1100</sup> Oxidation of certain aromatic thioureas produced vulcanization accelerators.<sup>1071</sup> Pinacolylthiourea is oxidised by permanganate to acetylurea.<sup>543</sup>

Diketene reacts with thioureas, guanidines, and S-substituted isothioureas to give products such as pyrimidines and thio-uracils.<sup>783</sup> Mono- or disubstituted derivatives of 2-aminothiazole may be prepared by condensation of an appropriately substituted thiourea with dimethyl chloroacetyl.<sup>703</sup> Isothiourea derivative,  $\text{NH}_2\text{C}(\text{SR}):\text{NH}$ , where R is alkyl, aryl, or alkaryl, reacts with acylating agents to give various products.<sup>513</sup>

Sulfathiourea has been prepared from a sulfonyl-s-benzylisothiourea, sulfur and sodium sulfide.<sup>313, 915</sup> Arylsulfonylthioureas have been prepared from an arylsulfonyl cyanamide and a thiol acid in the presence of an aliphatic monocarboxylic acid to form  $\text{ArSO}_2\text{NMC}(\text{S})\text{NHC}(\text{O})\text{R}$  (where M is H or a metal and R is alkyl) and then hydrolyzing to  $\text{ArSO}_2\text{NHC}(\text{S})\text{NH}_2$ .<sup>1386</sup>

The identity of Heilpern's pinacolylthiourea has been discussed.<sup>1116</sup>

Thiopegan derivatives have been prepared by refluxing certain ketones with substituted thioureas.<sup>1093</sup>

Thioureas are used in the preparation of thiobarbiturates. An example of such a preparation is found in the condensation of malonyl chloride with N,N'-diphenylthiourea.<sup>1155</sup> An example of the production of an imidazoline from the reaction of a thiourea with an alkyl halide is the reaction of ethylene thiourea with an alkyl halide to give 2-alkyl mercapto-2-imidazoline.<sup>45, 351</sup>

Diethyl allylthiourea and allylthiourea have been used as electron donors in the reduction of thionine with mercury light.<sup>999</sup>

### Physiological Effects on Plants

Thiourea is one of a number of chemicals that influence plant growth.<sup>1273</sup> It hastens the development of dormant buds in potatoes<sup>286</sup> and shortens the dormant period.<sup>287, 511, 598</sup> The number and weight of the sprouts are increased, but the difference disappears by end of summer.<sup>1324</sup> Thioureas increase the rate of streaming of the protoplasm,<sup>872</sup> the number of shoots,<sup>286</sup> and number of tubers but decrease their size.<sup>1404</sup> The starch content is decreased;<sup>286</sup> hollow heart is prevented.<sup>1375</sup> The protective property of thiourea against radiation-induced chromosome aberrations has been studied.<sup>905</sup>

Soaking sugar beet seed in thiourea solution does not increase tops or roots.<sup>340</sup> Soaking sweet potato roots in thiourea solution resulted in loss of proximal dominance.<sup>578</sup> The rest period of Jerusalem artichoke tubers is shortened.<sup>1239</sup>

Thiourea stimulates the germination of lettuce seed<sup>888, 1279, 1280</sup> but does not seem to alter the relationship between the root and the shoot.<sup>1241</sup> Thiourea and allylthiourea stimulate the seeds of *Striga lutea* to germinate.<sup>152</sup> Thiourea was the only one among 200 chemicals tried that caused the growth of two or more buds from a single eye of a potato.<sup>286</sup>

Soaking in thiourea solution hastened the sprouting of gladiolus seeds.<sup>1171</sup> Treatment with thiourea solution reduced both germination and tillering by a toxic rather than osmotic effect.<sup>448</sup>

Thiourea decreased the catalase content of cabbage leaves.<sup>788</sup> It inhibits photosynthesis and respiration in *Chlorella* cells.<sup>486</sup> The necrosis of *Gentiana* follows a peculiar course.<sup>568</sup> Thiourea poisons cucumber and potato oxidases,<sup>846</sup> and is lethal to *Avena Sativa*.<sup>987</sup> It can be used as a herbicide for Johnson grass and quack grass.<sup>1285</sup> Thiourea gave definite experimental control of stem-end rot and green wood decay in oranges.<sup>209</sup> Treatment of slices of apples, potatoes and the like with 0.1% solution of thiourea prevents or retards browning during drying.<sup>210, 286</sup> Thiourea affects oxidising enzymes.<sup>1427</sup> It influences the rate of hydrolysis of starch by amylase.<sup>219</sup>

A salt of dithioformamidine,  $\text{H}_2\text{N}(\text{NH}:)\text{CSSC}(:\text{NH})\text{NH}_2$ , added to flour improves the bread in several respects.<sup>145</sup> Thiourea is used in the production of green tea.<sup>1104</sup>

### Physiological Effects on Animals

The effects of thiourea on the thyroid gland have been extensively studied.<sup>28, 29, 69, 208, 211, 280, 301, 449, 479, 532, 588, 596a, 676, 796, 818, 875, 911, 1067c, 1200, 1242, 1311, 1330, 1340, 1392</sup> It and some of its derivatives inhibit the function of the thyroid.<sup>28, 577, 852, 925</sup> Thiourea<sup>30, 473, 1159</sup> and allylthiourea<sup>710</sup> cause thyroid enlargement and hyperplasia. Thiourea is useful in relieving thyrotoxicosis.<sup>562, 1063, 1391</sup> This has been attributed to its ability to react with iodine.<sup>180</sup> Methyl-, trimethyl- and tetramethyl-thiourea have been classed as strongly goitrogenic; thiourea, and its allyl and 1,3-diphenyl derivatives as weakly so.<sup>667</sup> Administering thiourea to an animal produces many of the effects of thyroidectomy.<sup>798, 800</sup> Thiourea affects the metabolism of iodine by the rat thyroid.<sup>713</sup> It inhibits the conversion of organic iodine to thyroxine.<sup>419</sup> It does not alter the thyroxine requirement of an animal but cuts down thyroxine synthesis.<sup>489</sup> Radioactive iodine, as sodium iodide, was injected into rats some of which had been dosed with thiourea. The thyroids of the normal rats fixed ten times as much of the iodine as the others.<sup>246</sup> Administering potassium iodide to rats protects them from the acute effects of subsequent doses of thiourea.<sup>488</sup> The intraperitoneal injection of 50 mg. of thiosinamine daily for 10 days caused marked hypertrophy of the thyroid in a rat without hypersecretion.<sup>1323</sup> Feeding allylthiourea along with 2-acetylaminofluorene caused the development of thyroid tumors.<sup>94</sup>

Thiourea was one of the nitrogen compounds considered in a study of metabolism.<sup>1013</sup> Allylthiourea raises body temperature of animals.<sup>868</sup>

The use of thiourea by aviators in high altitude flying has been recommended as a means of reducing thyroid activity. Rats fed on food containing thiourea were able to survive when the air pressure was reduced to 200 mm. corresponding to an altitude of 32,000 feet. This was attributed to their hypothyroidism.<sup>473, 479</sup> Autopsies of thiourea-fed rats revealed no pathological changes that could be attributed to thiourea.<sup>533b</sup> Large doses bring on rapid and fatal pulmonary edema in adult rats.<sup>853</sup> Young rats tolerate it better than old.<sup>297, 853</sup> A diet containing 1% of thiourea is fatal to chicks.<sup>1048</sup> The minimum lethal doses of S-methylisothiurea for dogs, cats, rabbits, and rats have been

determined.<sup>586</sup> Treatment of chick eggs at different stages of incubation with thiourea increased embryonic development 3 to 10 days beyond normal.<sup>1072</sup>

Various other studies have been made on the effects of thiourea on rats and other animals.<sup>27, 54, 62, 77, 207, 296, 314, 327, 328, 653, 689, 694, 745, 773, 782, 793, 827, 1056, 1175, 1240, 1313, 1318, 1372, 1407, 1429</sup>

S-Methyl-,<sup>388, 849, 1203</sup> S-isopropyl,<sup>388, 1029</sup> S-ethyl-, S-butyl- and S-*t*-amyl-isothiouras raise the blood pressure, slow the heart and stimulate respiration.<sup>388, 1332</sup> S-Methylisothiourea has been used in histamine shock.<sup>586</sup> Various thioureas and thio-pseudoureas have an effect on blood pressure.<sup>183, 1033, 1153, 1343</sup> The pharmacology of thiophenatine has been described.<sup>17</sup>

It has been found that thiourea-S<sup>35</sup> penetrates 3.5 mm. of the dentine of extracted teeth in approximately twenty minutes.<sup>1342</sup>

The hypnotic effects of 60 substituted thioureas have been compared. This increases with molecular weight in a homologous series, with a maximum with propyl amyl.<sup>277</sup> Allylthiourea and several others had a narcotic effect on frogs and rabbits.<sup>315</sup> Other studies have been made on the hypnotic properties of thiourea derivatives.<sup>158, 226, 698, 1202</sup> Thiourea retards the metamorphic process in tadpoles.<sup>479</sup> Its effects on metabolism in trypanosomes have been studied.<sup>468, 1192</sup>

The coagulation of blood is prevented by 3% of thiourea but only retarded by 4.5% of urea.<sup>953</sup> The rate of hemolysis of erythrocytes in various animals is influenced by thiourea.<sup>657</sup> Its antipyretic action is due to the thio group.<sup>556</sup>

Certain aromatic thiourea derivatives have been claimed as preventatives of contagious abortion in cattle.<sup>626</sup> Certain thiourea derivatives have been found to be strongly active against *log ascaris*.<sup>688</sup>

Thiourea has been used in measuring changes in body water.<sup>273</sup> It is excreted partly as such and partly oxidised.<sup>105, 880</sup> Only about 75% of it can be recovered.<sup>205b</sup> Ingested thiourea is distributed rapidly throughout the tissues and fluids of the body and appears in the urine within 30 minutes.<sup>1393</sup>

Other physiological effects including the effect of thiourea on cancer have been studied.<sup>15, 206, 289, 302, 325, 406, 426, 437, 458, 470, 474, 498, 537, 716, 755, 774, 813, 891, 906, 988, 1028, 1046, 1066, 1205, 1405</sup>

The response of the cortical layer of the *Arbacia* egg to thiourea is similar to that brought about by sperm cells.<sup>928</sup> Deca-

methylene-diisothiurea caused partial inhibition of the Pasteur effect.<sup>295</sup> Diethylaminoethyl thiourea and similar compounds have been investigated as substitutes for ergot.<sup>518</sup> A variety of dithiobiuret and isodithiobiuret derivatives possess activity against *Trypanosoma Congolense* when given subcutaneously to infected mice.<sup>381, 1406</sup>

Some improvement was observed in animals infected with human tuberculosis on injection of thiourea.<sup>160</sup> As a result, many studies have been made on the effects of thiourea and its derivatives on tuberculosis.<sup>34, 109, 149, 171, 173, 261, 358, 384c, 414, 455, 456, 516, 583, 585, 621, 686, 759, 869, 890, 993, 1124, 1327, 1328, 1329, 1369, 1411</sup> 4,4'-Diethoxythiocarbanilide has been shown to have high antitubercular activity in infected mice. The synthesis and testing of over 300 thiocarbanilides, 4,4'-RC<sub>6</sub>H<sub>4</sub>NHCSNHC<sub>6</sub>H<sub>4</sub>R', revealed the rather specific structural features necessary for antitubercular activity.<sup>172, 583</sup>

Thiourea derivatives have been tested as possible antimalarials.<sup>102, 255, 446, 506, 1082</sup>

The pharmacology<sup>847</sup> and pathology<sup>822</sup> of  $\alpha$ -naphthylthiourea have been studied. The pharmacological properties of various other thiourea derivatives have been tested.<sup>56, 103, 110, 168, 196, 225, 385, 387, 445, 457, 506, 654, 769, 847, 886, 887, 1029, 1174</sup> Bis-isothiuronium salts have been prepared and investigated as hypotensive agents.<sup>493</sup> The biological action of uracil-5-isothiuronium chloride, a new thymine antagonist, has been tested.<sup>62</sup>

The inhibitory effects of thiourea and its derivatives against the adverse effects of radiation have been studied.<sup>79, 312, 392, 522, 644, 744, 792</sup>

The effect of thioureas on blood parasites has been tested.<sup>71a, 71b, 322, 416, 1174</sup> A stabilized solution of 1-sulfanil-2-thiourea is said to be useful in otiatics.<sup>74</sup>

## Applications

### *As Antiseptics*

Only at concentrations above 1.25% is thiourea toxic to *B. lactis acidii*.<sup>1312</sup> It has a definite inhibitory action on pathogenic organisms.<sup>889, 953</sup> Its compounds with mercuric chloride and with other heavy metal salts are claimed as disinfectants.<sup>372</sup> Thio-

ureas of the stibanilic series have been used in the preparation of sterile therapeutic solutions for injections and as intermediates. High molecular weight alkyl-isothioureas are effective disinfectants.<sup>949</sup> Thiourea acts synergetically with sulfathiazole.<sup>812</sup> Certain thiuronium compounds exhibit bactericidal or bacteriostatic properties especially against gram-neg. organisms.<sup>108</sup>

### *As Pesticides*

Thiourea, its allyl and phenyl derivatives are toxic to mosquito larvae.<sup>181</sup> 1,3-Diphenylthiourea is useful in an insect spray or in dusting powders for poultry or domestic animals.<sup>524</sup> Combined with a copper salt, with or without arsenicals, it is recommended as a dusting powder.<sup>453</sup> Thiocarbanilide is said to be useful as a dusting powder for wheat to destroy rust.<sup>619</sup> Thiourea is toxic to flesh fly and to clothes moth larvae<sup>579, 857</sup> and serves as a moth repellent.<sup>922b</sup> Phenyl, allyl, and tolyl thioureas destroy the larvae of adult *Drosophila melanogaster*.<sup>215</sup> Thiourea is toxic to the larvae of *Musca domestica* and *Lucilia sericata*.<sup>579, 850</sup> Acyl derivatives of alkylthioureas are claimed as moth proofing agents.<sup>638</sup> Various diphenyl thioureas have been used to moth proof wool.<sup>883</sup> Thiourea or one of its substitution products, may be used with a protective colloid as an insecticide.<sup>1084</sup> They are destructive to all forms of insect life.<sup>524</sup> Thiourea dioxide has been used as a stabilizer for pesticides containing rotenone.<sup>1277</sup> Thioureas with a 1-alkyl substituent of at least eight carbons in length, and with a 3-substituent which contains a water-soluble polar group are excellent contact insecticides.<sup>1106</sup> 1-Allyl-3-(4-chloro-2-methylphenyl) thiourea is effective against Japanese beetle or Mexican jumping bean beetle.<sup>125</sup> Thiocarbamido-DDT is more effective than DDT against bedbugs.<sup>1341</sup> The effects of phenylthiocarbamide on the melanogenesis of amphibian larvae have been reported.<sup>761</sup> S-benzylisothiuronium dinitrophenates can be used as insecticides, fungicides, or molluscicides.<sup>250</sup> The toxicity to silk worms of a dust of allylthiourea and kaolin is low.<sup>463</sup>

Feeding tests with rabbits<sup>408</sup> and with rats<sup>533a</sup> show that thiourea is not harmful in the quantities that would be found on fruit which had been treated with it. The danger of cancer formation resulting from the presence of thiourea and other pesticide

residues in food has been reviewed.<sup>1295</sup> The biological properties of thiourea have been reviewed.<sup>1152</sup> It has been shown to be harmless as a 'fruit spray'.<sup>258</sup>

### *In Photography*

Thiourea has various uses in photography; with potassium ferricyanide as a reducer,<sup>659, 946</sup> in sepia toning,<sup>580, 659</sup> in the production of blue tones on gold chloride paper,<sup>580, 1207</sup> as a developer in mercury intensification<sup>659</sup> and as a fixing agent.<sup>659</sup> It may be used with hydroquinone as a developer.<sup>998</sup> Thiourea, its allyl, ethyl, diethyl and phenyl substitution products have been considered as substitutes for sodium thiosulfate in fixing baths but have not been found to be desirable.<sup>840</sup> Thiourea in alkaline solution is used in the toning of direct positive paper.<sup>424</sup> Thiosinamine may serve as a ripening agent.<sup>821</sup> Thiourea and some of its derivatives may also serve as ripening accelerators.<sup>378, 661, 982</sup> Thiourea dioxide has found use as a reducing agent in photography.<sup>1363</sup> Products obtained by cyclization of N-substituted N'-guanidinothiourea hydrochlorides are useful as stabilizers of photographic silver halide emulsions.<sup>433a</sup> Emulsions are sensitized by thiourea<sup>714, 835, 1039</sup> and allylthiourea.<sup>7, 187, 198, 835</sup> These and other thiourea derivatives find uses with photographically applicable dyes.<sup>682, 1206, 1298</sup> Diethylallylthiourea<sup>1016, 1364</sup> and allylphenylthiourea<sup>776</sup> may be employed in bleaching photographic dyes. Thiourea is used in photographic fog inhibitors.<sup>714</sup>

Thiourea or an arylthiourea is used in wall paint or enamel for rooms in which photographic emulsions are manufactured.<sup>1403</sup>

### *As Inhibitors*

Thiourea has maximum inhibiting effect on corrosion of iron by acids at 50 mg. per liter.<sup>55</sup> It decreases the rate of solution of aluminum in acid,<sup>1006</sup> of corrosion by pure water or by potassium chloride solution.<sup>24</sup> Various thiourea derivatives have also been studied in respect to altering the rate of attack of acids on steel and aluminum.<sup>362, 597, 865, 1283</sup> A substituted pseudo-thiourea serves as an inhibitor in acid solutions for cleaning and pickling metals.<sup>1164</sup> Thiosinamine, even in small concentrations, almost inhibits the autooxidation of linseed oil.<sup>1365</sup> Thiourea retards the drying of linseed and other like oils.<sup>669</sup> It has been suggested as an antioxidant for vitamin C.<sup>700</sup> It and its water soluble deriva-

tives are stabilizers for preparations containing vitamin C.<sup>469</sup> Combined with skim milk powder it is an antioxidant for fats.<sup>1394</sup> Thiamine and riboflavin are made less heat labile by thiourea.<sup>646</sup>

Thiourea inhibits the catalytic decomposition of hydrogen peroxide by finely divided silver.<sup>829</sup> Among the thioureas the aryl derivatives are the best as antioxidants in soap.<sup>1179</sup>

Thiourea and some of its derivatives are useful in polymerizations.<sup>1246</sup>

Thiocarbanilide prevents gum formation in light distillates<sup>1036</sup> and stabilizes cracked distillates.<sup>220</sup>

Thiourea and many of its derivatives are vulcanization accelerators.<sup>174, 311, 350, 510, 561, 786, 947, 1080, 1301, 1396</sup> Certain hydroxyphenyl and aminophenyl derivatives have been investigated as antioxidants in the aging of rubber.<sup>561</sup>

### *Miscellaneous Uses*

Thiourea has been suggested for many uses: for making molding powder,<sup>634</sup> for condensing with a lignin sulfonic acid,<sup>1057</sup> for impregnating in plywood manufacture with subsequent heating.<sup>832</sup> Its dimethyl derivative may be condensed to a plastic by heating.<sup>602</sup> Its S-lauryl, S-palmityl and S-stearyl derivatives are useful for increasing the plasticity of rubber.<sup>1067b</sup> Certain polythioureas from diamines have valuable fiber forming properties.<sup>336, 527</sup>

6-Amino-n-hexyldithiocarbamic acid,  $\text{NH}_2(\text{CH}_2)_6\text{NH}\cdot\text{CS}\cdot\text{SH}$ , is converted into a polymer by heating at  $200^\circ$  for 30 minutes. This can be spun into fibers.<sup>966</sup>

Thiourea may be condensed with imines.<sup>615</sup> It improves color fastness in dyeings on acetate rayon,<sup>517</sup> and cotton fibers swell in its solutions.<sup>697</sup> Thiourea and allyl thiourea have been used for selective adsorption of certain dye ions, to the exclusion of other dyes. The mechanism of this action is discussed.<sup>661</sup> S-Dodecylisothiurea<sup>590b</sup> and the condensation products of chloracetic esters with thiourea<sup>485</sup> are constituents of textile assistants. The same purpose is served by a product from thiourea and 2-hydroxy-5-chloromethylbenzoic acid,<sup>1222a</sup> and also by cetyl isothiuronium salts prepared from cetyl bromide and thiourea or its derivatives.<sup>655</sup> Other textile assistants from dichloromethyl ether and thiourea are known.<sup>214a</sup>

Surface-active agents can be obtained by the reaction of certain bis-halomethyl compounds with a thiourea.<sup>336</sup> Useful prod-



ucts are made by condensing aliphatic isothiocyanates with 1,2-alkylene imines.<sup>643</sup>

Thiourea increases the viscosity of shellac varnish.<sup>1326</sup> Of a number of derivatives of thiourea tested as rubber stabilizers, 1,3-bis (p-dimethylaminophenyl) thiourea gave the best protection against oxidation.<sup>561</sup> Other derivatives of thiourea, such as the mono- and dithiobiurets, are claimed to be useful as plasticizers, rubber accelerators, insecticides, and defoliant activators.<sup>476, 1067b, 1228a</sup>

Derivatives of thiourea containing at least one  $F_3C$  group and an  $SO_3H$  group are said to be useful in protecting wool, feathers, hair, leather, natural and synthetic fibers against moth worms and other textile pests.<sup>876</sup>

A substituted thiourea, such as thiocarbanilide, is said to be useful in flotation.<sup>997, 1182</sup> This may be combined with zinc cyanide or sulfonated.<sup>143</sup> The same claim is made for pinacolyl thiourea.<sup>929</sup>

Thiourea is recommended as a substitute for cyanide in silver plating bath.<sup>469</sup> In conjunction with caustic alkali it is a solvent for cellulose.<sup>823a</sup> It removes objectionable chlorine compounds from plastics.<sup>1213</sup>

In the presence of thiourea benzoyl chloride is catalytically reduced over platinum oxide to benzaldehyde almost quantitatively. It directs the hydrogenation of unsaturated ketones.<sup>1377</sup>

The use of thiourea in separating branch chain hydrocarbons has been previously discussed. A similarly suggested use has to do with the chemistry of fats, i.e. the separation of mixtures of triglycerides and fatty acids based on the fact that thiourea forms addition products with saturated and unsaturated fatty acids but not with triglycerides and oxidised fatty acids.<sup>919, 1131</sup> Thiourea complexes are also formed with certain petroleum hydrocarbons which can be separated by the use of this technique.<sup>1263, 1366</sup>

Certain thioureas have good adhesive properties.<sup>652</sup> Thiourea is said to improve the odor of thiocyanate insecticides.<sup>188</sup> Substituted thioureas,  $RR'NCSNH_2$ , have gained prominence as rodenticides. The most effective of these are N(1-naphthyl) thiourea,<sup>84, 326, 746, 1023, 1252</sup> and N(2-biphenyl) and N(4-biphenyl) thiourea.<sup>1056</sup> Thiuronium derivatives and thioimides have also been investigated as rodenticides.<sup>77</sup>

p-Fluorophenyl-, phenyl-p-fluorophenyl- and di-p-fluorophenyl thioureas are claimed as dye intermediates.<sup>837b</sup>

Thioureadioxide,  $(\text{NH}_2)_2\text{CSO}_2$ , is recommended as a reducing agent for changing vat dyes to their leuco forms,<sup>480, 771, 836</sup> and for stripping vat and azo dyes from fabrics.<sup>87, 836</sup> It has also been used to reduce aromatic nitro, azoxy, azo, and hydrazo compounds to amines and quinones to hydroquinones.<sup>480</sup> Other substituted thioureas are useful as intermediates for anthraquinone and other dyes.<sup>221, 1035</sup>

The use of thioureas in the preparation of various anesthetics, pharmaceuticals, and of certain industrially important chemicals has been reviewed.<sup>25, 960</sup> (p-Aminophenylsulfonyl) thiourea has been used for the preparation of pharmaceutical compounds.<sup>217, 1215</sup>

Thiourea or its derivatives have been of aid in the analysis of the following elements: cobalt,<sup>92, 1180</sup> copper,<sup>57, 92, 107, 274, 338, 968, 1110b, 1180, 1181, 1385</sup> nickel,<sup>92, 338, 1180, 1181</sup> selenium,<sup>671</sup> tellurium,<sup>671</sup> ruthenium,<sup>60, 1816</sup> osmium,<sup>60, 165, 274, 1110b, 1313</sup> zinc,<sup>338, 428</sup> bismuth,<sup>61, 92, 166, 454, 954, 1015</sup> lead,<sup>92, 338, 954, 1385</sup> cadmium,<sup>92, 274, 1110b, 1385</sup> phosphorus,<sup>898</sup> aluminum,<sup>908</sup> silver,<sup>92</sup> mercury,<sup>92, 429, 1385</sup> molybdenum,<sup>1417</sup> iron,<sup>1422</sup> and antimony.<sup>65</sup>

Thiourea has been used to increase the crystal size of various analytical precipitations.<sup>403</sup> The effect of thiourea on the electrocrystallization of copper has been described.<sup>15</sup> Thiourea and certain derivatives can be used to precipitate copper from homogeneous solutions.<sup>57, 1361</sup> It has been used for the colorimetric determination of phosphorus.<sup>898</sup>

Thiourea can be used in the electrolysis of water.<sup>1108</sup> It is also claimed to be a new ferroelectric.<sup>1225</sup>

### Thiourea Condensation Products

Urea and thiourea condense with aldehydes to form polymeric products some of which are important industrially. Of all the aldehydes formaldehyde is the one that gives the best products and finds the widest use. Urea-formaldehyde plastics are manufactured on a large scale. Those from thiourea are produced in smaller amounts but still are important. Both forms can be modified endlessly by additions of other components and by variations in the manufacturing processes. In many patents thiourea

is mentioned as an alternative to urea and in some a mixture of the two is specified.<sup>200, 523, 918, 921</sup> Thiourea is said to give more resistance to water<sup>1317</sup> and better wet strength to fibers coated with the condensation products.<sup>758</sup> Thiourea, though not expensive, does cost considerably more than urea. As its price comes down and as more experience in its applications is gained, its use will doubtless expand. One trouble with plastics containing thiourea is that they corrode the molds unless these are chromium plated.

In this brief section no attempt will be made to go into details as to processes. Some patents are grouped according to the conditions of the condensation and a number are listed without any attempt at classification.

Thiourea, condensed with acetaldehyde, gives a product of the composition,  $\text{H}_2\text{N}\cdot\text{CS}\cdot\text{N}:\text{CHCH}_3$ , which is decomposed by boiling water into the original constituents.<sup>1052b</sup> With aldehyde ammonia the product melts at  $183-4^\circ$  and has the composition  $\text{C}_5\text{H}_{11}\text{N}_3\text{S}$ .<sup>305a, 948b</sup> This has been formulated as  $\text{CH}_3\text{CHN}\cdot\text{CS}\cdot\text{NCHCH}_3\cdot\text{NH}_3$ .<sup>948b</sup> The compound from *i*-valeraldehyde ammonia melts at  $120-1^\circ$ .<sup>305a</sup> The product from thiourea and acetaldehyde, which melts at  $212^\circ$  and is insoluble, has been written also as the cyclic  $\text{SC}(\text{NH})_2\text{CHCH}_3$  but its properties indicate that it is polymeric.<sup>308c</sup>

Thiourea and chloral give anhydrodichloralthiourea,  $\text{Cl}_3\text{C}\cdot\text{CH}(\text{OH})\text{NH}\cdot\text{CS}\cdot\text{N}:\text{CHCCl}_3$ , m.  $131^\circ$ .<sup>393</sup> This can be reduced by zinc and acetic acid to tetrachlorodiethylthiourea, m.  $163^\circ$ .<sup>896</sup>

Useful products are obtained by condensing thiourea with benzaldehyde,<sup>765</sup> butyraldehyde,<sup>420</sup> acrolein,<sup>748</sup> crotonaldehyde,<sup>625, 977</sup> furfural,<sup>128, 692, 881</sup> and the aldehydes obtained by oxidising petroleum hydrocarbons.<sup>363</sup>

The condensation of formaldehyde with thiourea is already important and will probably become more so. Back in 1891 thiourea was treated with "chlormethyl alcohol," evidently a mixture of formalin and hydrochloric acid, and a solid obtained which had the composition  $\text{C}_2\text{H}_4\text{N}_2\text{S}$  and was regarded as methylenethiourea,  $\text{SC}(\text{NH})_2\text{CH}_2$ . It decomposed at  $180^\circ$  without melting. This was an unrecognized polymer.<sup>546</sup>

The kinetics and the constitution of the condensation reaction of thiourea with formaldehyde have been studied.<sup>815, 1236, 1238</sup>

Urea and thiourea are bifunctional and so are aldehydes, con-

sidered as methylene glycols,  $\text{HO}\cdot\text{CH}_2\cdot\text{OH}$  and  $\text{HO}\cdot\text{CHR}\cdot\text{OH}$ . This means that a chain once started continues to grow indefinitely, forming a linear polymer. If 12.7 g. of thiourea in 100 cc. of water and 12.5 g. of 40% formalin containing 0.07% formic acid are heated, the water evaporates leaving a syrup which solidifies to needles melting at  $97-8^\circ$ . This is monomethylolthiourea,  $\text{HOCH}_2\text{NH}\cdot\text{CS}\cdot\text{NH}_2$ . With more formaldehyde the dimethylol-,  $\text{HOCH}_2\text{NH}\cdot\text{CS}\cdot\text{NHCH}_2\text{OH}$ , m.  $86-8^\circ$ , is formed<sup>1017b</sup> Or two molecules of thiourea may be condensed with one of formaldehyde. A monoacylthiourea, treated with formaldehyde and conc. sulfuric acid at a low temperature, gives the methylene derivative which is saponified to methylenethiourea,  $\text{H}_2\text{N}\cdot\text{CS}\cdot\text{NHCH}_2\text{NH}\cdot\text{CS}\cdot\text{NH}_2$ , m.  $252^\circ$ .<sup>298, 299</sup> The low molecular condensation products are soluble in water but this solubility diminishes as the chain lengthens; the high polymers are insoluble in water. By conducting the condensation in a solvent in which methylolthiourea is only sparingly soluble it drops out as it is formed. In some other solvent dimethylolthiourea will separate out.<sup>1349d</sup> The dimethylolthiourea may be used as the starting material for further condensations.<sup>605, 637, 842, 1061</sup>

A resin is formed by heating thiourea and anhydrous polyformaldehyde in the absence of a solvent. Suitable catalysts and fillers may be added.<sup>49c, 51b, 379, 604, 705, 741, 1258</sup> The condensation may be effected at room temperature.<sup>49d</sup> Thiourea and phenol may be melted with dry formaldehyde to complete condensation.<sup>51a</sup> Thiourea and formaldehyde may be fused together with acetamide or benzamide.<sup>50</sup> Formaldehyde may be condensed with a hydroxyalkyl- or hydroxyaryl-thiourea.<sup>618</sup> The condensation may be stopped short of the final phase and the products so obtained mixed with additional formaldehyde and other ingredients and subjected to further treatment.<sup>1218</sup>

In the study of the constitution of urea-formaldehyde resins it has been found that methylene thiourea reacts with thiourea to give a stable tetramer containing one NH group per molecule.<sup>1238</sup> The condensation may be effected in two stages. Urea, or thiourea, may react with an excess up to two molecules of formaldehyde; thiourea, or urea may be added and the condensation completed.<sup>159, 363, 443, 444, 707, 768a, 1011, 1223, 1286</sup> The condensation may be carried out in solution in an alcohol,<sup>599, 639, 645, 1051, 1112, 1113</sup> one boiling above  $100^\circ$  as butyl, *i*-butyl, amyl, or benzyl.<sup>336,</sup>

<sup>357, 607</sup> The presence of an alcohol containing a haloaryl group,<sup>610</sup> chlorine or carboxyl,<sup>610</sup> or of one having more than eight carbon atoms,<sup>610, 620</sup> has been considered desirable.  $\alpha$ -Dichlorohydrin,<sup>823c</sup> an alcohol or mercaptan having 14 or more carbon atoms,<sup>485</sup> a mixture of an alcohol with a cyclic alcohol<sup>614</sup> or with Cello-solve,<sup>1111, 1142</sup> or with an ester of lactic acid,<sup>48, 1141, 1142</sup> and an alcohol and the mono-higher-fatty acid ester of a glycol<sup>630</sup> have been claimed as suitable media in which to effect the condensation. Other organic solvents may be used.<sup>637, 962, 1349c</sup>

Thiourea and formaldehyde may be heated with glycols<sup>842, 986</sup> or other polyhydric alcohols,<sup>606</sup> sorbitol,<sup>33</sup> sucrose,<sup>155</sup> starch,<sup>1191, 1282</sup> glue,<sup>1256</sup> cellulose,<sup>363</sup> or a cellulose derivative.<sup>559, 1230</sup> A thiourea formaldehyde condensation product is said to stabilize wet-processed starch pastes.<sup>1070</sup> Thiourea reacts with esters of mono- or poly-meric vinyl alcohol.<sup>203</sup>

Methylol amides of fatty acids,  $\text{RCONH}\cdot\text{CH}_2\text{OH}$ , may be used along with thiourea in the formation of resins;<sup>485, 976, 1220</sup> so may nitriles and amides.<sup>976, 1158, 1222e, 1224</sup>

Alcohols, mercaptans and aliphatic amides react with formaldehyde or its derivatives and the resulting compounds,  $\text{ROCH}_2\text{Cl}$ ,  $\text{RSCH}_2\text{Cl}$ ,  $\text{RCONHCH}_2\text{Cl}$  and  $(\text{RCONHCH}_2)_2\text{O}$  combined with thiourea.<sup>484, 485, 1219</sup> Urethans and thiourethans may be condensed with formaldehyde along with thiourea.<sup>49b, 624, 768a</sup> Di-thiourea,<sup>616</sup> nitrothiourea, guanylthiourea,<sup>148a</sup> thiobiuret, thio-semicarbazide<sup>1272</sup> and alkylthioureas<sup>972</sup> may be used along with urea or thiourea in making plastics. The thioureacuprous chloride complex may be used with formaldehyde in making plastics.<sup>1349a, 1351</sup> Ethylene oxide, other alkylene oxides<sup>126, 622</sup> or their polymers<sup>635</sup> and amines can be condensed with thiourea to form plastics.

Thioglycerol or 2-hydroxy-trimethylene-1,3-sulfide may be incorporated in a thiourea-formaldehyde mixture at any stage of the condensation.<sup>823b</sup> Sulfide linkages may be introduced into the polymers by adding hydrogen sulfide or ammonium sulfide.<sup>373, 380, 1004</sup>

The reaction product from aldehydeammonia and thiourea has been recommended as a vulcanization accelerator.<sup>276, 1169</sup> The same is said of one from a mercapto-aryl-thiazole, formaldehyde and thiourea.<sup>1396</sup>

The condensation products of ethylenethiourea with formaldehyde have been investigated.<sup>1237b</sup>

Some other patents on thiourea-formaldehyde condensation products are listed below without any attempt at classification or claim to completeness.<sup>3, 9, 46, 49a, 146, 147, 148b, 218, 267, 269, 323, 349, 352, 363, 436, 540, 627, 628, 632, 641, 643, 706, 707, 767, 777, 863, 892, 913, 922a, 933, 934, 943, 951, 962, 963, 1004, 1012, 1017a, 1019, 1020, 1027, 1061, 1073, 1078, 1081, 1119, 1127, 1140, 1157, 1221, 1249, 1287, 1357, 1376</sup>

### Physical Properties of Some Substituted Thioureas

#### THIOUREA

Thiourea, m.  $149^{\circ}$ ,<sup>335</sup>  $151^{\circ}$ ,<sup>68</sup>  $169^{\circ}$ ,<sup>186</sup>  $172^{\circ}$  (rhombic),<sup>901</sup>  $180^{\circ}$ ,<sup>528</sup>  $181.4^{\circ}$ ,<sup>1176</sup>  $184.5^{\circ}$ ,<sup>958</sup> Ac., m.  $163^{\circ}$ ,<sup>305a</sup>  $165^{\circ}$ ,<sup>752, 916, 1024</sup>  $166^{\circ}$ ,<sup>587b</sup> EtCO, m.  $148^{\circ}$ ,<sup>916</sup> *i*-PrCO, m.  $112^{\circ}$ ,<sup>308d</sup>  $114.5^{\circ}$ ,<sup>916</sup> BuCO, m.  $139^{\circ}$ ,<sup>916</sup> *i*-BuCO, m.  $157.5^{\circ}$ ,<sup>916</sup> AmCO, m.  $138^{\circ}$ ,<sup>916</sup> *i*-AmCO, m.  $155^{\circ}$ ,<sup>916</sup> Hex CO, m.  $133^{\circ}$ ,<sup>916</sup> HepCO, m.  $138^{\circ}$ ,<sup>916</sup> (Et)-(*i*-Am)CHCO, m.  $89.5^{\circ}$ ,<sup>916</sup> Dec CO, m.  $136.5^{\circ}$ ,<sup>916</sup> C<sub>11</sub>H<sub>23</sub>CO, m.  $138^{\circ}$ ,<sup>412</sup> C<sub>13</sub>H<sub>27</sub>CO, m.  $135^{\circ}$ ,<sup>412</sup> C<sub>15</sub>H<sub>31</sub>CO, m.  $136^{\circ}$ ,<sup>412</sup> C<sub>17</sub>H<sub>35</sub>CO, m.  $133^{\circ}$ ,<sup>412</sup> Oleoyl, m.  $113^{\circ}$ ,<sup>412</sup> HOOCCH<sub>2</sub>CH<sub>2</sub>CO, m.  $211^{\circ}$ ,<sup>1007</sup> Bz., m.  $132^{\circ}$ ,<sup>462</sup> Phthaloyl, m.  $172^{\circ}$  dec.,<sup>1009</sup> 5-Nitro-8-thiophenecarbonyl, m.  $206^{\circ}$ ,<sup>414</sup> 1,3-diAc., m.  $152^{\circ}$ ,<sup>752</sup> 1,3-Ac., Bz., m.  $105^{\circ}$ ,<sup>119, 308d</sup> 1,3-Ac., *p*-Ac., NHC<sub>6</sub>H<sub>4</sub>-SO<sub>2</sub>, m.  $202^{\circ}$  dec.,<sup>575</sup> PhSO<sub>2</sub>, m.  $138.8^{\circ}$ ,<sup>1395</sup>  $139^{\circ}$ ,<sup>25</sup> *m*-H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>SO<sub>2</sub>, m.  $166.5^{\circ}$ ,<sup>1328</sup> *p*-H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>SO<sub>2</sub>, m.  $149^{\circ}$ ,<sup>1184</sup>  $172^{\circ}$ ,<sup>574</sup>  $179^{\circ}$ ,<sup>385</sup>  $200^{\circ}$ ,<sup>25</sup> *p*-AcNHC<sub>6</sub>H<sub>4</sub>SO<sub>2</sub>, m.  $188^{\circ}$ ,<sup>1184</sup>  $192^{\circ}$ ,<sup>914</sup>  $195^{\circ}$ ,<sup>772</sup>  $198^{\circ}$  dec.,<sup>656</sup>  $199^{\circ}$ ,<sup>1395</sup>  $228^{\circ}$ .<sup>25</sup>

Nitrothiourea, m.  $134^{\circ}$ .<sup>983</sup>

#### THIOUREA COMPLEXES

- |            |   |
|------------|---|
| 1 Thiourea | •AcI, m. $106^{\circ}$ . <sup>308d</sup>  |
|            | •PrOCl, m. $117^{\circ}$ . <sup>308d</sup>  |
|            | •2-Bromopyridene-1-oxide, m. $160^{\circ}$ . <sup>1163</sup>                                |
| 2 Thiourea | •H <sub>2</sub> O <sub>2</sub> , m. $130^{\circ}$ . <sup>1315</sup>                         |
|            | •AgCl, m. $171^{\circ}$ , <sup>1052c, 1244</sup> •AgBr, m. $121^{\circ}$ . <sup>1052c</sup> |
|            | •NH <sub>4</sub> Br, m. $190^{\circ}$ . <sup>1374c</sup>                                    |
| 3 Thiourea | •Et <sub>2</sub> NH <sub>2</sub> Cl, m. $136^{\circ}$ . <sup>1052c</sup>                    |
|            | •Et <sub>2</sub> NH <sub>2</sub> Br, m. $134^{\circ}$ . <sup>1052c</sup>                    |
|            | •AgNO <sub>3</sub> , m. $141^{\circ}$ . <sup>1052c</sup>                                    |

- $\cdot\text{KCNS}$ , m.  $143^\circ$ .<sup>32</sup>  
 $\cdot\text{NH}_4\text{SCN}$ , m.  $144^\circ$ .<sup>32</sup>  
 4 Thiourea  $\cdot\text{NH}_4\text{Cl}$ , m.  $154^\circ$ ; <sup>1052c</sup>  $\cdot\text{NH}_4\text{Br}$ , m.  $174^\circ$ ; <sup>1052c</sup>  $\cdot\text{NH}_4\text{I}$ ,  
 m.  $186^\circ$ .<sup>1052c</sup>  
 $\cdot\text{CsI}$ , m.  $191^\circ$ ; <sup>32</sup>  $\cdot\text{RbI}$ , m.  $202^\circ$ .<sup>32</sup>  
 $\cdot\text{MeH}_3\text{NBr}$ , m.  $138^\circ$ .<sup>1052c</sup>  
 $\cdot\text{Et}_4\text{NBr}$ , m.  $160^\circ$ ; <sup>1052c</sup>  $\cdot\text{Et}_4\text{NI}$ , m.  $135^\circ$ .<sup>1052c</sup>

MONOSUBSTITUTED THIOUREAS,  $\text{RNHCSNH}_2$

R=Alkyl

- Methyl, m.  $120^\circ$ ,<sup>569d, 1105</sup>  $119^\circ$ ,<sup>281</sup>  $118^\circ$ ; <sup>12b, 223, 305a, 344</sup>  $\text{HCl}$ , m.  
 $87^\circ$ ; <sup>100</sup> 1-( $m\text{-NO}_2\text{C}_6\text{H}_4\text{CO}$ ), m.  $135^\circ$ ; <sup>1029</sup> 3-Ac., m.  $170.5^\circ$ ; <sup>916</sup>  
 3-*i*-PrCO, m.  $121.5^\circ$ ; <sup>916</sup> 3-BuCO, m.  $93^\circ$ ; <sup>916</sup> 3-*i*-BuCO, m.  
 $156^\circ$ ; <sup>916</sup> 3-AmCO, m.  $85^\circ$ ; <sup>916</sup> 3-Hex CO, m.  $76^\circ$ ; <sup>916</sup> 3-HepCO,  
 m.  $81.5^\circ$ ; <sup>916</sup> 3-Dec CO, m.  $80.5^\circ$ ; <sup>916</sup> 3,3-diAc., m.  $108^\circ$ .<sup>973</sup>  
 Ethyl, m.  $89^\circ$ ,<sup>569b</sup>  $106^\circ$ ,<sup>569b</sup>  $111^\circ$ ,<sup>730, 1025</sup>  $113^\circ$ ,<sup>423, 569d</sup>  $144^\circ$ ; <sup>344</sup>  
 d  $1.0030$ .<sup>467</sup>  
 Propyl, m.  $111^\circ$ ,<sup>344</sup>  $110^\circ$ .<sup>541</sup>  
*i*-Propyl, m.  $157^\circ$ .<sup>660</sup>  
 Butyl, m.  $78^\circ$ ,<sup>344</sup>  $79^\circ$ .<sup>569b, 569c</sup>  
*i*-Butyl, m.  $90^\circ$ ,<sup>466</sup>  $91^\circ$ ,<sup>344, 570</sup>  $93.5^\circ$ ,<sup>569b</sup>  $98^\circ$ ; <sup>439a, 439c</sup> b.  $160^\circ$ .<sup>344</sup>  
*s*-Butyl, m.  $128.5^\circ$ ,<sup>305a, 305c</sup>  $135^\circ$ ,<sup>569c</sup>  $136^\circ$ ,<sup>439c, 935</sup>  $137^\circ$ ; <sup>439a, 439c,</sup>  
<sup>1306</sup> D m.  $137^\circ$ ; <sup>104, 439a, 1306</sup>  $[\alpha]_D + 22.09$ ,<sup>1306</sup>  $[\alpha]_D^{20} + 22.77$  to  
 $22.93$ ; <sup>439a, 439c, 439d</sup>  $[M]_D^{20} + 30.06$  to  $+30.27$ .<sup>439a</sup>  
*t*-Butyl, m.  $165^\circ$ ,<sup>1338</sup>  $172^\circ$ .<sup>1136</sup>  
 Amyl, m.  $75^\circ$ ,<sup>344</sup>  $93^\circ$ .<sup>569d</sup>  
*i*-Amyl, m.  $91^\circ$ ,<sup>305a, 305c</sup>  $92^\circ$ ,<sup>344</sup>  $93^\circ$ .<sup>569d</sup>  
 Hexyl, m.  $83^\circ$ .<sup>344, 421</sup>  
*i*-Hexyl, m.  $62^\circ$ .<sup>687</sup>  
 Heptyl, m.  $90^\circ$ .<sup>344</sup>  
 Octyl, m.  $114^\circ$  dec.<sup>660</sup>  
*s*-Octyl, m.  $112.5^\circ$ .<sup>660</sup>  
*t*-Octyl, m.  $85^\circ$ .<sup>121</sup>  
 Decyl, m.  $104^\circ$ .<sup>494</sup>  
 Dodecyl, m.  $107^\circ$ .<sup>579</sup>  
 Cetyl, m.  $75^\circ$ ,<sup>1115</sup>  $114^\circ$ .<sup>596b</sup>  
 Heptadecyl, m.  $108^\circ$ ,<sup>596b</sup>  $111^\circ$ .<sup>1300</sup>  
 Allyl, m.  $74^\circ$ ,<sup>1269</sup>  $75^\circ$ ,<sup>1069</sup>  $78^\circ$ ; <sup>1374a</sup> d  $78.1$   $1.1100$ ; <sup>355</sup>  $n_D$   $1.59362$ ; <sup>355</sup>

1-Ac., m.  $96^{\circ}$ ; <sup>308a</sup> 3-Ac., m.  $74^{\circ}$ ; <sup>308a</sup> 3-NCCH<sub>2</sub>CO-, m.  $139^{\circ}$ ; <sup>984</sup>  
 3-[*p*-(*p*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>)C<sub>6</sub>H<sub>4</sub>SO<sub>2</sub>], m.  $176^{\circ}$ .<sup>585</sup>  
 Crotyl, m.  $64^{\circ}$ ,<sup>1198</sup>  $66^{\circ}$ ,<sup>734</sup>  $85^{\circ}$ ,<sup>569c</sup>  $92.5^{\circ}$ .<sup>1156</sup>  
 CH<sub>2</sub>=CHCH<sub>2</sub>CH<sub>2</sub>, m.  $66^{\circ}$ ,<sup>725</sup>  $67^{\circ}$ .<sup>374</sup>  
 CH<sub>2</sub>=CHCH(Me), m.  $88.5^{\circ}$ ,<sup>738</sup>  $110^{\circ}$ .<sup>734</sup>  
 Cyclohexyl, m.  $162^{\circ}$ ; <sup>1199</sup> 3-Ac., m.  $105^{\circ}$ .<sup>736</sup>

## R=Substituted Alkyl

HOCH<sub>2</sub>, m.  $98^{\circ}$ .<sup>1017b</sup>  
 Glucose, decomposes at  $216^{\circ}$ ; <sup>401.5</sup>  $[\alpha]_D^{70}$   $-35.5$ .<sup>401.5</sup>  
 EtOCH<sub>2</sub>, m.  $93^{\circ}$ .<sup>674</sup>  
 MeCOCH<sub>2</sub>, HCl, m.  $126^{\circ}$ .<sup>992</sup>  
 BrCH<sub>2</sub>CH<sub>2</sub>, m.  $174.2^{\circ}$ .<sup>879</sup>  
 BrCH<sub>2</sub>CHBrCH<sub>2</sub>, m.  $139^{\circ}$ ,<sup>439b</sup>  $140.5^{\circ}$ .<sup>305d</sup>  
 ICH<sub>2</sub>CHICH<sub>2</sub>, m.  $130.5^{\circ}$  dec.,<sup>439b</sup>  $133.5^{\circ}$ .<sup>305d</sup>  
 NC, m.  $135^{\circ}$ .<sup>873</sup>  
 HOOCCH<sub>2</sub>, m.  $171^{\circ}$  dec.<sup>753.5</sup>  
 MeS(CH<sub>2</sub>)<sub>3</sub>, m.  $67^{\circ}$ ; <sup>728</sup> sulfone, m.  $116^{\circ}$  <sup>1143</sup>  $117^{\circ}$ .<sup>732</sup>  
 MeS(CH<sub>2</sub>)<sub>4</sub>, m.  $45^{\circ}$ ; <sup>1145</sup> sulfone, m.  $144^{\circ}$ ,<sup>1109</sup>  $147^{\circ}$ .<sup>732</sup>  
 MeS(CH<sub>2</sub>)<sub>5</sub>, m.  $67.5^{\circ}$ .<sup>731</sup>

## R=Aryl

Phenyl, m.  $154^{\circ}$ ,<sup>642, 901, 978, 1174</sup>  $157^{\circ}$ ; <sup>277</sup> 1-Bz., m.  $124.5^{\circ}$ ; <sup>600</sup>  
 3-(*m*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CO), m.  $154^{\circ}$ .<sup>1029</sup>  
 PhCH<sub>2</sub>, m.  $82.5^{\circ}$ ,<sup>370</sup>  $101^{\circ}$ ,<sup>990</sup>  $162^{\circ}$ ,<sup>744</sup>  $163^{\circ}$ .<sup>1103</sup>  
 PhCHMe, m.  $106^{\circ}$ .<sup>970</sup>  
 PhCH<sub>2</sub>CH<sub>2</sub>, m.  $137^{\circ}$ .<sup>134</sup>  
 Ph(CH<sub>2</sub>)<sub>3</sub>, m.  $111^{\circ}$ .<sup>134</sup>  
 Ph(CH<sub>2</sub>)<sub>4</sub>, m.  $95^{\circ}$ .<sup>134</sup>  
 Ph<sub>2</sub>CH, m.  $184^{\circ}$ ,<sup>272</sup>  $185^{\circ}$ .<sup>702</sup>  
 PhCH<sub>2</sub>PhCH, m.  $171.5^{\circ}$ .<sup>701</sup>  
 Ph<sub>3</sub>C, m.  $212^{\circ}$ ,<sup>137</sup>  $317^{\circ}$ .<sup>900</sup>  
 C<sub>10</sub>H<sub>7</sub>,  $\alpha$  m.  $184^{\circ}$ ,<sup>877</sup>  $190^{\circ}$ ,<sup>1107</sup> 3-(*m*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CO), m.  $181^{\circ}$ ; <sup>1209</sup>  
 $\beta$  m.  $180^{\circ}$ ,<sup>1390</sup> 3-(*m*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CO), m.  $195^{\circ}$ .<sup>1299</sup>  
 $\alpha$ -C<sub>10</sub>H<sub>7</sub>CH<sub>2</sub>, m.  $124^{\circ}$ .<sup>1102</sup>

## R=Substituted Phenyl

ClC<sub>6</sub>H<sub>4</sub>, *o*, m.  $143^{\circ}$ ,<sup>394</sup>  $145^{\circ}$ ; <sup>1297</sup> *m*, m.  $138^{\circ}$ ; <sup>394</sup> *p*, m.  $183^{\circ}$ ,<sup>1250</sup>  
 $175^{\circ}$ ,<sup>1174</sup>  $139^{\circ}$ ; <sup>1103</sup> 3-(*m*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CO-), m.  $161^{\circ}$ .<sup>1029</sup>



- $\text{Cl}_2\text{C}_6\text{H}_3$ , 2,3-, m.  $148^\circ$ ; <sup>343</sup> 2,4-, m.  $158^\circ$ , <sup>201</sup>  $156^\circ$ ; <sup>394</sup> 3,4-, m.  $198^\circ$ ; <sup>394</sup> 2,5-, m.  $194^\circ$ .<sup>394</sup>  
 5,2-(Cl)(MeO) $\text{C}_6\text{H}_3$ , m.  $133^\circ$ , <sup>153</sup>  $145^\circ$ .<sup>368</sup>  
 $\text{BrC}_6\text{H}_4$ , o, m.  $125^\circ$ ; <sup>343</sup> p, m.  $183^\circ$ ; <sup>1174</sup> 3-(*m*- $\text{O}_2\text{NC}_6\text{H}_4\text{CO}$ ), m.  $176^\circ$ ; <sup>1029</sup> 3-[*p*-(*p*- $\text{O}_2\text{NC}_6\text{H}_4$ ) $\text{C}_6\text{H}_4\text{SO}_2$ ], m.  $92^\circ$ .<sup>585</sup>  
 $\text{Br}_2\text{C}_6\text{H}_3$ , 2,4-, m.  $170^\circ$ , <sup>201</sup>  $171^\circ$ ; <sup>195</sup> 2,5-, m.  $130^\circ$ .<sup>343</sup>  
 3,4- $\text{BrMeC}_6\text{H}_3$ , 3-(*m*- $\text{O}_2\text{NC}_6\text{H}_4\text{CO}$ ), m.  $183^\circ$ .<sup>1299</sup>  
 $\text{IC}_6\text{H}_4$ , o, m.  $157^\circ$ ; <sup>343</sup> m, m.  $160^\circ$  dec.; <sup>343</sup> p, 3-[*p*-(*p*- $\text{O}_2\text{NC}_6\text{H}_4$ )- $\text{C}_6\text{H}_4\text{SO}_2$ ], m.  $186^\circ$ .<sup>1185</sup>  
 $\text{FC}_6\text{H}_4$ , m, m.  $116^\circ$ ; <sup>153</sup> p, m.  $164^\circ$ .<sup>345</sup>  
 $\text{HOC}_6\text{H}_4$ , o, m.  $161^\circ$ ; <sup>1174</sup> m, m.  $176^\circ$ ; <sup>319</sup>  $183^\circ$ , <sup>686</sup>  $287^\circ$ ; <sup>342</sup> 3-(*p*- $\text{ClC}_6\text{H}_4\text{CO}$ -), m.  $210^\circ$  dec.<sup>1105</sup> p, m.  $214^\circ$ .<sup>1174</sup>  
 2,6-(HO) $_2\text{C}_6\text{H}_3$ , dec.  $203^\circ$ .<sup>1174</sup>  
 3,4-(HO)(HOOC) $\text{C}_6\text{H}_3$ , Me ester decomposes at  $183^\circ$ ; <sup>686</sup> Et ester, m.  $183^\circ$  dec.<sup>686</sup>  
 2,5-(HO)(MeOOC) $\text{C}_6\text{H}_3$ , m.  $163^\circ$ .<sup>1174</sup>  
 2,4-(HO)(NCS) $\text{C}_6\text{H}_3$ , m. 138 dec.<sup>688</sup>  
 $\text{MeOC}_6\text{H}_4$ , o, m.  $149^\circ$ , <sup>343</sup>  $152^\circ$ , <sup>1174</sup>  $156^\circ$ , <sup>277</sup>, <sup>1297</sup>  $158^\circ$ ; <sup>494</sup> m, m.  $159^\circ$ , <sup>750</sup>  $160^\circ$ , <sup>856</sup>  $166^\circ$ ; <sup>277</sup> p, m.  $204^\circ$ , <sup>1102</sup>  $208^\circ$ , <sup>300</sup>  $211^\circ$ , <sup>342</sup>  $218^\circ$ .<sup>277</sup>  
 (MeO) $_2\text{C}_6\text{H}_3$ , 2,5-, m.  $160^\circ$ ; <sup>343</sup> 3,4-, m.  $234^\circ$ .<sup>856</sup>  
 $\text{EtOC}_6\text{H}_4$ , o, m.  $126^\circ$ , <sup>343</sup>  $127^\circ$ ; <sup>277</sup> m, m.  $176^\circ$ , <sup>277</sup>  $167^\circ$ ; <sup>343</sup> p, m.  $176^\circ$ , <sup>342</sup>  $175^\circ$ , <sup>277</sup>  $173^\circ$ , <sup>642</sup>  $171^\circ$ , <sup>941</sup>  $165^\circ$ .<sup>300</sup>  
 3-Bz., m.  $168.5^\circ$ ; <sup>941</sup> 3-(*p*- $\text{H}_2\text{NC}_6\text{H}_4\text{CO}$ -), m.  $183^\circ$ ; <sup>941</sup> 3-(*p*- $\text{AcNHC}_6\text{H}_4\text{CO}$ -) m.  $190.5^\circ$ ; <sup>941</sup> 3-(*p*- $\text{Me}_2\text{NC}_6\text{H}_4\text{CO}$ -), m.  $182^\circ$ .<sup>941</sup>  
 4,2-(EtO)( $\text{O}_2\text{N}$ ) $\text{C}_6\text{H}_3$ , m.  $177^\circ$ .<sup>343</sup>  
*p*- $\text{Et}_2\text{NCH}_2\text{CH}_2\text{OC}_6\text{H}_4$ , m.  $101^\circ$ .<sup>110</sup>, <sup>626</sup>  
 2,5-(PrO)( $\text{O}_2\text{N}$ ) $\text{C}_6\text{H}_3$ , m.  $192^\circ$ .<sup>1026</sup>  
*p*-Oct $\text{OC}_6\text{H}_4$ , m.  $112^\circ$ .<sup>1415</sup>  
*p*- $\text{C}_{12}\text{H}_{25}\text{OC}_6\text{H}_4$ , m.  $127^\circ$ .<sup>1415</sup>  
*p*- $\text{C}_{14}\text{H}_{29}\text{OC}_6\text{H}_4$ , m.  $129^\circ$ .<sup>1415</sup>  
*p*- $\text{C}_{16}\text{H}_{33}\text{OC}_6\text{H}_4$ , m.  $129.5^\circ$ .<sup>1415</sup>  
*p*- $\text{C}_{17}\text{H}_{35}\text{OC}_6\text{H}_4$ , m.  $130^\circ$ .<sup>1415</sup>  
*p*-Ph $\text{OC}_6\text{H}_4$ , m.  $184^\circ$ .<sup>791</sup>  
*p*-[3,5- $\text{Me}_2\text{C}_6\text{H}_3\text{O}$ ] $\text{C}_6\text{H}_4$ , m.  $151^\circ$ .<sup>636</sup>  
 $\text{H}_2\text{NC}_6\text{H}_4$ , o, m.  $168^\circ$ ; <sup>1174</sup> p, m.  $190^\circ$ , <sup>1174</sup>  $202^\circ$ ; <sup>446</sup> *p*- $\text{AcNHC}_6\text{H}_4$ , m.  $212^\circ$ .<sup>446</sup>  
*p*- $\text{Me}_2\text{NC}_6\text{H}_4$ , m.  $181^\circ$ , <sup>1356</sup>  $190^\circ$ .<sup>343</sup>  
*p*- $\text{Et}_2\text{NC}_6\text{H}_4$ , m.  $167^\circ$ .<sup>1356</sup>

- $\text{CH}_2(\text{CH}_2\text{CH}_2)_2\text{NC}_6\text{H}_4$ , m.  $189.5^\circ$ .<sup>1356</sup>  
 $\text{O}(\text{CH}_2\text{CH}_2)_2\text{NC}_6\text{H}_4$ , m.  $228^\circ$  dec.<sup>1356</sup>  
 $\text{O}_2\text{NC}_6\text{H}_4$ , *o*, m.  $136^\circ$ ; <sup>23</sup>  $137^\circ$ ; <sup>361</sup> 3-(*m*- $\text{O}_2\text{NC}_6\text{H}_4\text{CO}$ -), m.  $185^\circ$ ; <sup>1299</sup> *m*, m.  $160^\circ$ ; <sup>1174</sup>  $158^\circ$ ; <sup>750</sup> 3-(*m*- $\text{O}_2\text{NC}_6\text{H}_4\text{CO}$ -), m.  $195^\circ$ ; <sup>1299</sup> *p*, m.  $190^\circ$ ; <sup>342</sup> 3-(*m*- $\text{O}_2\text{NC}_6\text{H}_4\text{CO}$ -), m.  $179^\circ$ ; <sup>1299</sup> 3-(*p*- $\text{O}_2\text{NC}_6\text{H}_4\text{CO}$ -), m.  $189^\circ$ .<sup>941</sup>  
 $\text{HOOC}_6\text{H}_4$ , *o*, 3-(*m*- $\text{O}_2\text{NC}_6\text{H}_4\text{CO}$ ), m.  $205^\circ$ ; <sup>1299</sup> 3-(*p*- $\text{MeC}_6\text{H}_4\text{CO}$ ), m.  $240^\circ$  dec.; <sup>417</sup> *p*, 3-(*m*- $\text{O}_2\text{NC}_6\text{H}_4\text{CO}$ ), m.  $222^\circ$ ; <sup>1299</sup> *o*, Me ester, m.  $124^\circ$ ; <sup>1174</sup> Et ester, m.  $121^\circ$ ; <sup>1174</sup>  $305^\circ$ ; <sup>343</sup>  $308^\circ$ ; <sup>1102</sup> *m*, Et ester, m.  $136^\circ$ ; <sup>750, 1325</sup>  $295^\circ$ ; <sup>343</sup> *p*, Et ester, m.  $160^\circ$ ; <sup>1325</sup>  $159^\circ$ .<sup>343</sup>  
3,4-( $\text{HOOC}$ ) $_2\text{C}_6\text{H}_3$ , decomposes at  $179^\circ$ .<sup>384c</sup>  
2,3-( $\text{HO}$ )( $\text{HOOC}$ ) $\text{C}_6\text{H}_3$ , 3-Bz., m.  $220^\circ$  dec.<sup>417</sup>  
3,4-( $\text{HO}$ )( $\text{HOOC}$ ) $\text{C}_6\text{H}_3$ , 3-Bz., m.  $200^\circ$  dec.<sup>417</sup>  
4,3-( $\text{HO}$ )( $\text{HOOC}$ ) $\text{C}_6\text{H}_3$ , 3-(*p*- $\text{ClC}_6\text{H}_4\text{CO}$ -), m.  $240^\circ$  dec.<sup>417</sup>  
 $\text{NCC}_6\text{H}_4$ , *m*, m.  $137^\circ$ ; <sup>343</sup> *p*, m.  $169^\circ$ .<sup>343</sup>  
2,5-( $\text{NC}$ ) $_2\text{C}_6\text{H}_3$ , m.  $228^\circ$ .<sup>343</sup>  
*p*- $\text{NCSC}_6\text{H}_4$ , m.  $127^\circ$ .<sup>688</sup>  
*p*- $\text{MeCOC}_6\text{H}_4$ , m.  $215^\circ$  dec.<sup>343</sup>  
*p*- $\text{HOOC}_6\text{H}_4\text{NHSO}_2\text{C}_6\text{H}_4$ , m.  $218^\circ$ .<sup>360</sup>  
*p*-(2-Thiazolyl- $\text{NHSO}_2$ ) $\text{C}_6\text{H}_4$ , m.  $217^\circ$  dec.<sup>1410</sup>  
*p*-(2-Pyridyl- $\text{NHSO}_2$ ) $\text{C}_6\text{H}_4$ , m.  $216^\circ$  dec.<sup>1410</sup>  
*p*-[2-(3,4-Dimethyl)-Isoxazolyl- $\text{NHSO}_2$ ] $\text{C}_6\text{H}_4$ , m.  $187^\circ$  dec.<sup>1410</sup>  
*p*-(2-Pyrimidyl  $\text{NHSO}_2$ ) $\text{C}_6\text{H}_4$ , m.  $230^\circ$  dec.<sup>1410</sup>

## R=Miscellaneous Aryl

- $\text{MeC}_6\text{H}_4$ , *o*, m.  $158^\circ$ ; <sup>431</sup>  $160^\circ$ ; <sup>978</sup>  $161^\circ$ ; <sup>1174</sup>  $164^\circ$ ; <sup>277, 324</sup> *m*, m.  $111^\circ$ ; <sup>1174</sup>  $119^\circ$ ; <sup>277</sup> *p*, m.  $186^\circ$ ; <sup>642</sup>  $191^\circ$ ; <sup>277</sup> 3-(*m*- $\text{O}_2\text{NC}_6\text{H}_4\text{CO}$ ), *o*, m.  $185^\circ$ ; <sup>1299</sup> *m*, m.  $127^\circ$ ; <sup>1299</sup> *p*, m.  $159^\circ$ ; <sup>1299</sup> 3-(*p*- $\text{O}_2\text{NC}_6\text{H}_4\text{CO}$ ), *p*, m.  $163^\circ$ ; <sup>941</sup> 3-(*p*- $\text{H}_2\text{NC}_6\text{H}_4\text{CO}$ ), *p*, m.  $193^\circ$ ; Ac., m.  $203.5^\circ$ .<sup>941</sup>  
3,4- $\text{ClMeC}_6\text{H}_3$ , m.  $138^\circ$ .<sup>153</sup>  
5,2- $\text{BrMeC}_6\text{H}_3$ , m.  $186^\circ$ .<sup>342</sup>  
3,4-( $\text{H}_2\text{N}$ ) $\text{MeC}_6\text{H}_3$ , m.  $172^\circ$ .<sup>1174</sup>  
( $\text{O}_2\text{N}$ ) $\text{MeC}_6\text{H}_3$ , 3,2-, m.  $171^\circ$ ; <sup>341</sup> 4,2-, m.  $140^\circ$ ; <sup>341</sup> 5,2-, m.  $188.5^\circ$ ; <sup>342</sup> 2,4-, m.  $122^\circ$ ; <sup>341</sup> 3,4-, m.  $138^\circ$ ; <sup>341</sup> 3-(*m*- $\text{O}_2\text{NC}_6\text{H}_4\text{CO}$ ), m.  $175^\circ$ .<sup>1299</sup>  
*p*- $\text{EtC}_6\text{H}_4$ , m.  $138^\circ$ .<sup>153</sup>  
 $\text{Me}_2\text{C}_6\text{H}_3$ , 2,3-, m.  $182^\circ$ ; <sup>343</sup> 2,4-, m.  $185^\circ$ ; <sup>342</sup> 2,5-, m.  $135.5^\circ$ ; <sup>342</sup> 2,6-, m.  $190^\circ$ ; <sup>343</sup> 3,4-, m.  $142.5^\circ$ .<sup>342</sup>

- 4,5,2-(MeO)<sub>2</sub>PrC<sub>6</sub>H<sub>2</sub>, m. 193°. <sup>1281</sup>  
 4-*i*-PrC<sub>6</sub>H<sub>4</sub>, m. 134°. <sup>153</sup>  
 2,4,5-Me<sub>3</sub>C<sub>6</sub>H<sub>2</sub>, m. 168°. <sup>342</sup>  
 3,2,4,6-(O<sub>2</sub>N)Me<sub>3</sub>C<sub>6</sub>H, m. 199°. <sup>342</sup>  
*p*-PhC<sub>6</sub>H<sub>4</sub>, m. 190°. <sup>70</sup>  
 ClC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>, *o*, m. 126°; <sup>1167</sup> *m*, m. 133°; <sup>1167</sup> *p*, m. 141°. <sup>1167</sup>  
 BrC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>, *o*, m. 127°; <sup>1167</sup> *m*, m. 151°; <sup>1167</sup> *p*, m. 157°. <sup>1167</sup>  
 IC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>, *m*, m. 151°; <sup>1167</sup> *p*, m. 164°. <sup>1167</sup>  
 MeOC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>, *o*, m. 122°; <sup>729</sup> *m*, m. 100°; <sup>375</sup> *p*, m. 135°, <sup>1102</sup>  
 138.5°. <sup>727</sup>  
*p*-HOC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>CH<sub>2</sub>, m. 198°. <sup>596b</sup>  
 2-[1-BrC<sub>10</sub>H<sub>8</sub>], m. 204°. <sup>593</sup>  
 1-[8-MeOC<sub>10</sub>H<sub>8</sub>], m. 187°. <sup>245, 1306</sup>  
 1-[4-NCSC<sub>10</sub>H<sub>8</sub>], m. 250°. <sup>688</sup>

R=Heterocyclic

- 6-Quinolyl, m. 218°. <sup>644</sup>  
 8-Chloro-5-quinolyl, m. 211°. <sup>642</sup>  
 2-Carbazyl, m. 278°. <sup>642</sup>  
 9-Ethyl-3-carbazyl, m. 210°. <sup>642</sup>  
 4-Indazyl, m. 226°. <sup>642</sup>  
 Nicotinyl, m. 210°. <sup>320</sup>  
 2-Pyridyl, m. 148°; <sup>381</sup> 3-Bz., m. 144°. <sup>381</sup>  
 2-Thiazolyl·HCl, m. 204°. <sup>89</sup>  
 2-(4-Me) Thiazolyl, m. 175°. <sup>89</sup>  
 COOEt·HCl, m. 117°. <sup>992</sup>

### 1,1-DISUBSTITUTED THIOUREAS, RR'NCSNH<sub>2</sub>

R=Methyl

R'

- Methyl, m. 82°, <sup>1229</sup> 155°, <sup>1025, 1105</sup> 159°. <sup>1347</sup>  
*i*-Propyl, m. 102°. <sup>257</sup>  
 Phenyl, m. 105°, <sup>277</sup> 107°; <sup>1347</sup> 3-Bz., m. 137°. <sup>381</sup>  
 PhCH<sub>2</sub>, m. 148.5°. <sup>401</sup>  
 MeOC<sub>6</sub>H<sub>4</sub>, *o*, m. 104°; <sup>277</sup> *m*, m. 145°; <sup>277</sup> *p*, m.  
 148°. <sup>277</sup>  
 EtOC<sub>6</sub>H<sub>4</sub>, *o*, m. 97°; <sup>277</sup> *m*, m. 111°; <sup>277</sup> *p*, m. 144°. <sup>277</sup>  
 MeC<sub>6</sub>H<sub>4</sub>, *o*, m. 109°; <sup>277</sup> *m*, m. 76°; <sup>277</sup> *p*, m. 112°. <sup>277</sup>  
 β-C<sub>10</sub>H<sub>7</sub>, m. 170°. <sup>593</sup>

## R=Ethyl

R'

Ethyl, m. 102°; <sup>1025, 1347</sup> 170°; <sup>1229</sup> 3-Ac., m. 152°.<sup>1101</sup>Phenyl, m. 103.5°; <sup>374</sup> 113°.<sup>277</sup>PhCH<sub>2</sub>, m. 123°; <sup>513</sup> 123.5°.<sup>401, 690</sup>MeOC<sub>6</sub>H<sub>4</sub>, *o*, m. 118°; <sup>277</sup> *m*, m. 128° <sup>277</sup> *p*, m. 140°.<sup>277</sup>EtOC<sub>6</sub>H<sub>4</sub>, *o*, m. 99°; <sup>277</sup> *m*, m. 152°; <sup>277</sup> *p*, m. 106°.<sup>277</sup>MeC<sub>6</sub>H<sub>4</sub>, *o*, m. 93°; <sup>277</sup> *m*, m. 108°; <sup>277</sup> *p*, m. 101°.<sup>277</sup>EtC<sub>6</sub>H<sub>4</sub>, *o*, m. 71°; <sup>158</sup> *p*, m. 103°.<sup>158</sup> $\alpha$ -C<sub>10</sub>H<sub>7</sub>, m. 158°.<sup>642</sup>R=C<sub>3</sub>H<sub>7</sub>

R

R'

Propyl,

Propyl, m. 67°.<sup>1347</sup>Phenyl, m. 108°; <sup>277</sup> 105°; <sup>381</sup> 3-Bz., m. 127°.<sup>381</sup>MeOC<sub>6</sub>H<sub>4</sub>, *o*, m. 85°; <sup>277</sup> *m*, m. 153°; <sup>277</sup> *p*, m. 122°.<sup>277</sup>EtOC<sub>6</sub>H<sub>4</sub>, *o*, m. 124°; <sup>277</sup> *m*, m. 173°; <sup>277</sup> *p*, m. 103°.<sup>277</sup>MeC<sub>6</sub>H<sub>4</sub>, *o*, m. 87°; <sup>277</sup> *m*, m. 115°; <sup>277</sup> *p*, m. 78°.<sup>277</sup>*i*-Propyl,*p*-EtC<sub>6</sub>H<sub>4</sub>, m. 104°.<sup>158</sup>R=C<sub>4</sub>H<sub>9</sub>

R

R'

Butyl,

Ph, m. 89°.<sup>277</sup>MeOC<sub>6</sub>H<sub>4</sub>, *o*, m. 94°; <sup>277</sup> *m*, m. 106°; <sup>277</sup> *p*, m. 129°.<sup>277</sup>EtOC<sub>6</sub>H<sub>4</sub>, *o*, m. 126°; <sup>277</sup> *m*, m. 126°; <sup>277</sup> *p*, m. 78°.<sup>277</sup>MeC<sub>6</sub>H<sub>4</sub>, *o*, m. 68°; <sup>277</sup> *m*, m. 66°; <sup>277</sup> *p*, m. 98°.<sup>277</sup>*t*-Butyl,Phenyl, m. 119.5°.<sup>374</sup>R=C<sub>5</sub>H<sub>11</sub>

R

R'

Amyl,

Amyl, m. 64°.<sup>1347</sup>Phenyl, m. 78°.<sup>277</sup>MeOC<sub>6</sub>H<sub>4</sub>, *o*, m. 80°; <sup>277</sup> *m*, m. 85°; <sup>277</sup> *p*, m. 139°.<sup>277</sup>EtOC<sub>6</sub>H<sub>4</sub>, *o*, m. 122°; <sup>277</sup> *m*, m. 56°; <sup>277</sup> *p*, m. 57°.<sup>277</sup>MeC<sub>6</sub>H<sub>4</sub>, *o*, m. 73°; <sup>277</sup> *p*, m. 78°.<sup>277</sup>*i*-Amyl,*i*-Amyl, m. 209°.<sup>1229</sup>

## R=Miscellaneous Alkyl

R

R'

CH<sub>2</sub>:CHCH<sub>2</sub>CH<sub>2</sub>,Phenyl, m. 57°.<sup>374</sup>

| R  | R'   |
|--|--|
| MeCH:CHCH <sub>2</sub> ,   | Phenyl, <i>cis</i> , m. 110.5°; <sup>374</sup> <i>trans</i> , m. 86°. <sup>374</sup> |
| Cyclohexyl,  | PhCH <sub>2</sub> , m. 94°. <sup>401</sup>   |
| Me <sub>2</sub> NCH <sub>2</sub> CH <sub>2</sub> ,                 | PhCH <sub>2</sub> , m. 105°; <sup>425</sup> 107°. <sup>1227</sup>                    |
|  | <i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> , m. 146°. <sup>425</sup>                 |
|  | <i>p</i> -EtOC <sub>6</sub> H <sub>4</sub> , m. 138°. <sup>425</sup>                 |
|  | <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> , m. 130°. <sup>425</sup>                  |
| Piperidinoethyl,   | Phenyl, m. 115°. <sup>425</sup>  |
| Me <sub>2</sub> NCH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> , | PhCH <sub>2</sub> , m. 102°. <sup>425</sup>  |
| Piperidinopropyl,  | Phenyl, m. 112°. <sup>425</sup>  |
| MeOOC,   | MeOOC, m. 118°; <sup>307</sup> picrate, m. 151°. <sup>307</sup>                      |

R=Aryl

| R  | R'  |
|--|---|
| Phenyl,                                      | Phenyl, m. 210° dec.; <sup>989</sup> 212°; <sup>642</sup> 218°; <sup>72</sup> 226°; <sup>780b</sup> |
|  | 3-Ac., m. 141°; <sup>308a</sup> ; 3-Bz., m. 135°; <sup>308a</sup>                                   |
|  | 3-( <i>m</i> -O <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> CO), m. 195°. <sup>1299</sup>           |
|  | β-C <sub>10</sub> H <sub>7</sub> , m. 224° dec. <sup>989</sup>                                      |
| PhCH <sub>2</sub> ,                          | PhCH <sub>2</sub> , m. 140°; <sup>1347</sup> 157°. <sup>990</sup>                                   |
|  | <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> , m. 155.5°. <sup>1355</sup>                              |
| <i>p</i> -HOC <sub>6</sub> H <sub>4</sub> ,  | <i>p</i> -HOC <sub>6</sub> H <sub>4</sub> , m. 232° dec. <sup>989</sup>                             |
| <i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> , | β-C <sub>10</sub> H <sub>7</sub> , m. 216° dec. <sup>989</sup>                                      |
| <i>o</i> -MeC <sub>6</sub> H <sub>4</sub> ,  | 4,2-(O <sub>2</sub> N)MeC <sub>6</sub> H <sub>3</sub> , m. 154°. <sup>341</sup>                     |

## 1,3-DISUBSTITUTED THIOUREAS RNHCSNHR'

R=Methyl

| R'                   |   |
|----------------------|---|
| Methyl,              | m. 50°; <sup>1105</sup> 52°. <sup>305b, 541</sup>   |
| MeOCH <sub>2</sub> , | m. 77°. <sup>1134</sup>   |
| EtOCH <sub>2</sub> , | m. 83.4°. <sup>1134</sup>   |
| Ethyl,               | m. 54°. <sup>569b</sup>   |
| Propyl,              | m. 78.5°. <sup>1135</sup>   |
| <i>i</i> -Propyl,    | m. 100°. <sup>985</sup>   |
| <i>i</i> -Butyl,     | m. 77.5°. <sup>541</sup>  |
| <i>s</i> -Butyl,     | m. 80°; <sup>305b</sup> 84°; <sup>1306</sup> [α] <sub>D</sub><br>+30.49°. <sup>1306</sup> |
| <i>t</i> -Butyl,     | m. 111°. <sup>1135</sup>  |
| <i>i</i> -Amyl,      | m. 76°. <sup>305b</sup>   |
| <i>t</i> -Octyl,     | m. 108°. <sup>481</sup>   |

R'

|   |  |
|---|--|
| CH <sub>2</sub> :CHCH <sub>2</sub> ,  | m. 56°. <sup>12a</sup>   |
| Phenyl,   | m. 112.5°, <sup>288</sup> 113°. <sup>1389</sup>  |
| <i>p</i> -BrC <sub>6</sub> H <sub>4</sub> ,   | m. 148°. <sup>594</sup>  |
| 3,4-(HO)(HOOC)C <sub>6</sub> H <sub>3</sub> ,   | decomposes at 192°. <sup>34</sup>  |
| 3,4-(HOOC) <sub>2</sub> C <sub>6</sub> H <sub>3</sub> ,   | decomposes at 180°. <sup>384c</sup>  |
| <i>p</i> -H <sub>2</sub> NSO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> ,                           | m. 196°. <sup>1327</sup>   |
| <i>m</i> -(4-Chloro-2-pyrimidyl)<br>NH <sub>2</sub> SO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> , | m. 195.5°. <sup>759</sup>  |
| <i>p</i> -PhC <sub>6</sub> H <sub>4</sub> ,   | m. 142°. <sup>150</sup>  |
| C <sub>10</sub> H <sub>7</sub> ,  | α m. 192°, <sup>344</sup> , <sup>1262</sup> 198°; <sup>346</sup> β m.<br>127°, <sup>150</sup> 130°. <sup>593</sup> |
| Phenylamidino,  | HCl, m. 117°. <sup>780b</sup>  |

R=Ethyl

R'

|   |  |
|---|--|
| Ethyl,  | m. 77°. <sup>569b</sup> , <sup>647</sup>   |
| HOCH <sub>2</sub> CH <sub>2</sub> ,           | m. 75°. <sup>1025</sup>  |
| <i>i</i> -Butyl,                              | m. 77.5°. <sup>305b</sup>  |
| <i>s</i> -Butyl,                              | m. 58°, <sup>305b</sup> 67°; <sup>1306</sup> [α] <sub>D</sub> + 25.06. <sup>1306</sup>                             |
| <i>i</i> -Amyl,                               | m. 46°. <sup>305b</sup>  |
| EtCH(OH)CHMe,                                 | m. 105°. <sup>663</sup>  |
| Me <sub>2</sub> CCH <sub>2</sub> CH(Me)OH,    | m. 198.5°. <sup>683</sup>  |
| CH <sub>2</sub> :CHCH <sub>2</sub> ,          | m. 47°, <sup>984</sup> 54°. <sup>12a</sup>   |
| Phenyl,                                       | m. 99°, <sup>902</sup> 102°, <sup>1389</sup> 107°. <sup>288</sup>  |
| <i>p</i> -BrC <sub>6</sub> H <sub>4</sub> ,   | m. 129°. <sup>594</sup>  |
| 3,4-(HO)(HOOC)C <sub>6</sub> H <sub>3</sub> , | m. 184°. <sup>34</sup>   |
| <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> ,   | m. 99°. <sup>757</sup>   |
| <i>p</i> -PhC <sub>6</sub> H <sub>4</sub> ,   | m. 165°. <sup>150</sup>  |
| C <sub>10</sub> H <sub>7</sub> ,              | α m. 118°, <sup>344</sup> 121°; <sup>346</sup> , <sup>1262</sup> β m.<br>142°, <sup>150</sup> 155°. <sup>757</sup> |
| 2-Tetrahydronaphthyl,                         | m. 131.5°. <sup>1359</sup>   |

R=C<sub>3</sub>H<sub>7</sub>

R

R'

|         |   |
|---------|---|
| Propyl, | Propyl, m. 71°. <sup>541</sup>  |
|         | <i>i</i> -Propyl, m. 76°, <sup>150</sup> 91°. <sup>1134</sup> , <sup>1135</sup>     |
|         | <i>s</i> -Butyl, m. 53°; <sup>1306</sup> [α] <sub>D</sub> + 22.296. <sup>1306</sup> |
|         | <i>t</i> -Butyl, m. 57.5°. <sup>1135</sup>  |
|         | Cyclohexyl, m. 89.5°. <sup>1134</sup>   |

| R                 | R'  |
|-------------------|---|
|                   | Phenyl, m. $62^{\circ}$ , <sup>288</sup> $64^{\circ}$ . <sup>1389</sup>   |
|                   | MeOCH <sub>2</sub> , m. $59.5^{\circ}$ . <sup>1134</sup>  |
|                   | 3,4-(HO)(HOOC)C <sub>6</sub> H <sub>3</sub> , m. $182^{\circ}$ . <sup>34</sup>  |
|                   | O <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> , <i>o</i> , m. $168^{\circ}$ ; <sup>341</sup> <i>m</i> , m. $119^{\circ}$ ; <sup>341</sup> <i>p</i> , m. $176^{\circ}$ . <sup>341</sup>                              |
|                   | (O <sub>2</sub> N)MeC <sub>6</sub> H <sub>3</sub> , 4,2-, m. $173^{\circ}$ ; <sup>341</sup> 5,2-, m. $157^{\circ}$ ; <sup>341</sup> 2,4-, m. $147^{\circ}$ ; <sup>341</sup> 3,4-, m. $144^{\circ}$ . <sup>341</sup> |
|                   | <i>p</i> -PhC <sub>6</sub> H <sub>4</sub> , m. $156^{\circ}$ . <sup>150</sup>   |
|                   | C <sub>10</sub> H <sub>7</sub> , $\alpha$ m. $67^{\circ}$ , <sup>347</sup> $102^{\circ}$ , <sup>346</sup> $103^{\circ}$ ; <sup>1262</sup> $\beta$ m. $114^{\circ}$ . <sup>150</sup>                                 |
| <i>i</i> -Propyl, | <i>i</i> -Propyl, m. $141.5^{\circ}$ , <sup>1134</sup> $161^{\circ}$ . <sup>660</sup>   |
|                   | <i>s</i> -Butyl, m. $112.5^{\circ}$ ; <sup>1306</sup> $[\alpha]_D + 21.12$ . <sup>1306</sup>  |
|                   | <i>t</i> -Butyl, m. $149.5^{\circ}$ . <sup>1135</sup>   |
|                   | Dodecyl, m. $75.5^{\circ}$ . <sup>1134</sup>  |
|                   | Cyclohexyl, m. $140^{\circ}$ . <sup>1134</sup>  |
|                   | Phenyl, m. $102^{\circ}$ . <sup>288</sup>   |
|                   | $\alpha$ -C <sub>10</sub> H <sub>7</sub> , m. $143^{\circ}$ . <sup>1262</sup>   |
|                   | MeOCH <sub>2</sub> , m. $81.5^{\circ}$ . <sup>1134</sup>  |
|                   | EtOCH <sub>2</sub> , m. $78^{\circ}$ . <sup>1134</sup>  |
|                   | Me <sub>2</sub> NCH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> , m. $80^{\circ}$ ; <sup>1134</sup> , <sup>1368</sup> picrate m. $159^{\circ}$ . <sup>1134</sup>   |
|                   | <i>p</i> -ClC <sub>6</sub> H <sub>4</sub> , m. $125^{\circ}$ . <sup>394</sup>   |
|                   | <i>p</i> -AcNHC <sub>6</sub> H <sub>4</sub> , m. $209^{\circ}$ , <sup>447</sup> $212^{\circ}$ . <sup>446</sup>  |
|                   | <i>p</i> -H <sub>2</sub> NSO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> , m. $194^{\circ}$ . <sup>1327</sup>  |
|                   | 3-(5-Chloro-2-pyrimidylsulfonyl)C <sub>6</sub> H <sub>4</sub> , m. $195^{\circ}$ dec. <sup>759</sup>  |
|                   | 2-Pyridyl, m. $130^{\circ}$ . <sup>1082</sup>   |
|                   | 6-Methoxy-8-quinolyl, m. $137.5^{\circ}$ . <sup>887</sup>   |



| R      | R'   |
|--------|--|
| Butyl, | Butyl, m. $65^{\circ}$ . <sup>1133</sup>   |
|        | <i>s</i> -Butyl, m. $32^{\circ}$ ; <sup>1306</sup> $[\alpha]_D + 19.54$ . <sup>1306</sup>  |
|        | Cyclohexyl, m. $81^{\circ}$ . <sup>1137</sup>  |
|        | Phenyl, m. $63^{\circ}$ , <sup>1389</sup> $65^{\circ}$ . <sup>592</sup>  |
|        | <i>p</i> -BrC <sub>6</sub> H <sub>4</sub> , m. $111^{\circ}$ . <sup>594</sup>  |
|        | 3,4-(HO)(HOOC)C <sub>6</sub> H <sub>3</sub> , m. $179^{\circ}$ . <sup>34</sup>   |
|        | <i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> , m. $74^{\circ}$ . <sup>182</sup>  |
|        | <i>p</i> -AcNHC <sub>6</sub> H <sub>4</sub> , m. $186^{\circ}$ . <sup>446</sup>  |
|        | <i>p</i> -PhC <sub>6</sub> H <sub>4</sub> , m. $155^{\circ}$ . <sup>150</sup>  |
|        | C <sub>10</sub> H <sub>7</sub> , $\alpha$ m. $98^{\circ}$ , <sup>308a</sup> $100^{\circ}$ , <sup>346</sup> $109^{\circ}$ ; <sup>1262</sup> $\beta$ m. $119^{\circ}$ . <sup>150</sup> |

| R                | R'   |
|------------------|--|
| <i>i</i> -Butyl, | <i>i</i> -Butyl, m. 88°. <sup>305b</sup><br>CH <sub>2</sub> :CHCH <sub>2</sub> , m. 28.5°. <sup>541</sup><br>Phenyl, m. 82°. <sup>305b, 541</sup><br><i>p</i> -BrC <sub>6</sub> H <sub>4</sub> , m. 119°. <sup>594</sup><br><i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> , m. 112°. <sup>182</sup><br><i>p</i> -PhC <sub>6</sub> H <sub>4</sub> , m. 157°. <sup>150</sup><br>C <sub>10</sub> H <sub>7</sub> , α m. 177°; <sup>308a</sup> β m. 137°. <sup>150</sup> |
| <i>s</i> -Butyl, | Phenyl, m. 101°. <sup>305b</sup><br><i>p</i> -AcNHC <sub>6</sub> H <sub>4</sub> , m. 214°. <sup>446</sup><br>α-C <sub>10</sub> H <sub>7</sub> , m. 137°. <sup>1262</sup>   |
| <i>t</i> -Butyl, | <i>t</i> -Butyl, m. 150° dec. <sup>1135</sup> 162°, <sup>1338</sup> 163°. <sup>1338</sup><br><i>t</i> -Octyl, m. 100°. <sup>121</sup><br>Cyclohexyl, m. 157°. <sup>1135</sup><br>Phenyl, m. 119°. <sup>121</sup><br>Me <sub>2</sub> NCH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> , m. 49.5°. <sup>1135</sup>   |

R=C<sub>5</sub>H<sub>11</sub>

| R                         | R'   |
|---------------------------|--|
| Amyl,                     | Phenyl, m. 69°, <sup>1389</sup> 71°. <sup>592</sup><br><i>p</i> -BrC <sub>6</sub> H <sub>4</sub> , m. 115°. <sup>594</sup><br><i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> , m. 82.5°. <sup>182</sup><br><i>p</i> -MeC <sub>6</sub> H <sub>4</sub> , m. 217°. <sup>1247</sup><br><i>p</i> -PhC <sub>6</sub> H <sub>4</sub> , m. 147°. <sup>150</sup><br>C <sub>10</sub> H <sub>7</sub> , α m. 103°, <sup>347, 1262</sup> 104°; <sup>346</sup><br>β m. 114°. <sup>150</sup> |
| <i>i</i> -Amyl,           | <i>i</i> -Amyl, m. 73°. <sup>305b</sup><br>Phenyl, m. 102°. <sup>305b, 1389</sup><br>PhCH <sub>2</sub> , m. 110°. <sup>134</sup><br><i>p</i> -BrC <sub>6</sub> H <sub>4</sub> , m. 105°. <sup>594</sup><br><i>p</i> -PhC <sub>6</sub> H <sub>4</sub> , m. 130°. <sup>150</sup><br>C <sub>10</sub> H <sub>7</sub> , α m. 92°, <sup>347</sup> 95°, <sup>346</sup> 97.5°; <sup>1262</sup><br>β m. 116°. <sup>150</sup>  |
| Me <sub>2</sub> CHCH(Me), | α-C <sub>10</sub> H <sub>7</sub> , m. 134°. <sup>1262</sup>  |

## R=Higher Alkyl

| R      | R'  |
|--------|---|
| Hexyl, | Hexyl, m. 40°, <sup>421</sup> 46°. <sup>687</sup><br>Phenyl, m. 104°. <sup>592</sup><br><i>p</i> -BrC <sub>6</sub> H <sub>4</sub> , m. 189°. <sup>594</sup> |



| R                 | R'   |   |
|-------------------|--|---|
|                   | $\alpha$ -C <sub>10</sub> H <sub>7</sub> ,         | m. 79°. <sup>1262</sup> 89°. <sup>347</sup>   |
| <i>i</i> -Hexyl   | MeOCH <sub>2</sub> ,                               | m. 37°. <sup>1134</sup>   |
|                   | $\alpha$ -C <sub>10</sub> H <sub>7</sub> ,         | m. 79.5°. <sup>1262</sup>   |
| Heptyl,           | Phenyl,  | m. 71°. <sup>592</sup>  |
|                   | <i>p</i> -BrC <sub>6</sub> H <sub>4</sub> ,        | m. 156°. <sup>594</sup>   |
|                   | <i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> ,       | m. 89°. <sup>182</sup>  |
|                   | <i>p</i> -PhC <sub>6</sub> H <sub>4</sub> ,        | m. 149°. <sup>150</sup>   |
|                   | C <sub>10</sub> H <sub>7</sub> ,                   | $\alpha$ m. 62°. <sup>347</sup> 66°. <sup>346</sup> 69°. <sup>1262</sup> $\beta$<br>m. 115°. <sup>150</sup> |
| <i>s</i> -Heptyl, | $\alpha$ -C <sub>10</sub> H <sub>7</sub> ,         | m. 102°. <sup>1262</sup>  |
| Octyl,            | $\alpha$ -C <sub>10</sub> H <sub>7</sub> ,         | m. 72.2°. <sup>1262</sup>   |
| <i>s</i> -Octyl,  | $\alpha$ -C <sub>10</sub> H <sub>7</sub> ,         | m. 83°. <sup>1262</sup>   |
| <i>t</i> -Octyl,  | Me <sub>2</sub> NCH <sub>2</sub> CH <sub>2</sub> , | m. 86°. <sup>121</sup>  |
| Dodecyl,          | Me(CH <sub>2</sub> ) <sub>11</sub> ,               | m. 70°. <sup>579</sup>  |
| Tetradecyl,       | Tetradecyl,  | m. 81°. <sup>878</sup>  |
|                   | Phenyl,  | m. 78°. <sup>878</sup>  |
| Cetyl,            | Cetyl,   | m. 88°. <sup>878</sup>  |
|                   | Phenyl,  | m. 82.5°. <sup>878</sup>  |
| Heptadecyl,       | Phenyl,  | m. 79°. <sup>1300</sup>   |

## R=Unsaturated

| R                                    | R'   |   |
|--------------------------------------|--|---|
| CH <sub>2</sub> :CHCH <sub>2</sub> , | HOCH <sub>2</sub> CH <sub>2</sub>  | m. 78.5°. <sup>1133</sup>                               |
|                                      | Diacetonyl,  | m. 138°. <sup>1289</sup>                                |
|                                      | Cyclohexyl,  | m. 72°. <sup>1133</sup>                                 |
|                                      | 3,4-(HO)(HOOC)C <sub>6</sub> H <sub>3</sub> ,  | m. 173° dec., <sup>1185</sup><br>180°. <sup>34</sup>    |
|                                      | <i>p</i> -H <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> ,  | m. 120°; <sup>445</sup> HCl, m.<br>230°. <sup>445</sup> |
|                                      | <i>m</i> -AcNHC <sub>6</sub> H <sub>4</sub> ,  | m. 175°. <sup>445</sup>                                 |
|                                      | 3,4-(HOOC) <sub>2</sub> C <sub>6</sub> H <sub>3</sub> ,  | decomposes at 184°. <sup>384c</sup>                     |
|                                      | <i>p</i> -H <sub>2</sub> NSO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> ,  | m. 182°; <sup>445</sup> 184°. <sup>1327</sup>           |
|                                      | <i>p</i> -Me <sub>2</sub> NSO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> ,   | m. 143°. <sup>445</sup>                                 |
|                                      | <i>p</i> -( <i>p</i> -H <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> )NH <sub>2</sub> SO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> , | m. 181° dec. <sup>445</sup>                             |
|                                      | <i>m</i> -(5-Chloro-2-pyrimidylsulfonyl)C <sub>6</sub> H <sub>4</sub> ,  | m.<br>186°. <sup>759</sup>                              |
|                                      | <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> ,  | m. 107°. <sup>734</sup>                                 |
|                                      | <i>m</i> -F <sub>3</sub> CC <sub>6</sub> H <sub>4</sub> ,  | m. 94.5°. <sup>162</sup>                                |
|                                      | 4,5,2-(MeO) <sub>2</sub> PrC <sub>6</sub> H <sub>2</sub> ,   | m. 154°. <sup>1281</sup>                                |

| R  | R'   |
|--|--|
|  | HOOCCH:CHC <sub>6</sub> H <sub>4</sub> , <i>m</i> , <i>m</i> . 177°; <sup>445</sup> <i>p</i> , <i>m</i> . 171°. <sup>445</sup> |
|  | Methyl-2-thiazolyl, 4-, <i>m</i> . 180°; <sup>1185</sup> 5-, <i>m</i> . 178°. <sup>432</sup>                                   |
|  | 5-Benzylthiomethyl-2-oxazoliny, <i>m</i> . 100°. <sup>432</sup>  |
|  | 5-Anilino-3-(1,2,4-triazolyl), <i>m</i> . 133°. <sup>432</sup>   |
|  | 5-Benzothio-3-(1,2,4-triazolyl), <i>m</i> . 116°. <sup>432</sup>   |
|  | 3-Benzylthio-1-phenyl-5-(1,2,4-triazolyl), <i>m</i> . 138°. <sup>432</sup>   |
|  | 5-Benzylthiomethyl-1-phenyl-3-(1,2,4-triazolyl), <i>m</i> . 129°. <sup>432</sup>   |
|  | 5-Methylthio-1-phenyl-3-(1,2,4-triazolyl), <i>m</i> . 138°. <sup>432</sup>   |
|  | COOEt, <i>m</i> . 57°. <sup>459</sup>  |
| MeCH:CHCH <sub>2</sub> ,                             | Phenyl, <i>m</i> . 110°. <sup>734</sup>  |
|  | <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> , <i>m</i> . 125°. <sup>734</sup>  |
| CH <sub>2</sub> :CHCH <sub>2</sub> CH <sub>2</sub> , | Phenyl, <i>m</i> . 105.5°. <sup>734</sup>  |
|  | <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> , <i>m</i> . 82°. <sup>734</sup>   |
| CH <sub>2</sub> :CHCHMe,                             | Cyclohexyl, <i>m</i> . 112.5°. <sup>1133</sup>   |
|  | Phenyl, <i>m</i> . 81°. <sup>734</sup> 87°. <sup>733</sup>   |
|  | <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> , <i>m</i> . 61°. <sup>733</sup>   |
|  | α-C <sub>10</sub> H <sub>7</sub> , <i>m</i> . 120.5°. <sup>733</sup>   |
| CH <sub>2</sub> :CMeCH <sub>2</sub> ,                | <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> , <i>m</i> . 67°. <sup>734</sup>   |
| Oleyl,   | Oleyl, <i>m</i> . 69°. <sup>1339</sup>   |

R=Cyclic

| R           | R'  |
|-------------|---|
| Cyclohexyl, | Cyclohexyl, <i>m</i> . 181°. <sup>1199</sup>  |
|             | MeOCH <sub>2</sub> , <i>m</i> . 104°. <sup>1134</sup>   |
|             | EtOCH <sub>2</sub> , <i>m</i> . 110°. <sup>1134</sup>   |
|             | Phenyl, <i>m</i> . 148°. <sup>182</sup> 151°. <sup>1199</sup>   |
|             | PhCH <sub>2</sub> , <i>m</i> . 94°. <sup>401</sup>  |
|             | 3,4-(HO)(HOOC)C <sub>6</sub> H <sub>3</sub> , <i>m</i> . 189°. <sup>34</sup>  |
|             | <i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> , <i>m</i> . 135°. <sup>182</sup>  |
|             | <i>p</i> -Et <sub>2</sub> NCH <sub>2</sub> CH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , 2HCl, decomposes at 212°. <sup>110, 626</sup> |
|             | <i>p</i> -PhC <sub>6</sub> H <sub>4</sub> , <i>m</i> . 180°. <sup>150</sup>   |
|             | C <sub>10</sub> H <sub>7</sub> , α <i>m</i> . 142°; <sup>1262</sup> β <i>m</i> . 172°. <sup>150</sup>                                     |

| R                                  | R'  |
|------------------------------------|---|
| <i>p</i> -Ethoxy- <i>c</i> -hexyl, | <i>p</i> -PhCH <sub>2</sub> CH <sub>2</sub> , m. 119°. <sup>583</sup>       |
| 2-Carbethoxy- <i>c</i> -hexyl,     | 2-Carbethoxy- <i>c</i> -hexyl, m. 133°. <sup>356</sup>                      |
| Methyl- <i>c</i> -hexyl,           | Methyl- <i>c</i> -hexyl, m. 119°. <sup>135</sup>                            |
|                                    | Phenyl, m. 92°. <sup>135</sup>  |
| Bornyl,                            | Bornyl, m. 228°; <sup>1420</sup> [α] <sub>D</sub><br>-19.4. <sup>1420</sup> |

## R=Substituted Alkyl

| R  | R'  |
|--|---|
| HOCH <sub>2</sub> ,                                  | HOCH <sub>2</sub> , m. 88°. <sup>1017b</sup>  |
| Cl <sub>2</sub> CHCH <sub>2</sub> ,                  | Cl <sub>2</sub> CHCH <sub>2</sub> , m. 163°. <sup>896</sup>   |
| Cl <sub>3</sub> CCH:                                 | Cl <sub>3</sub> CCH:, m. 151°. <sup>896</sup>   |
| HOCH <sub>2</sub> CH <sub>2</sub> ,                  | HOCH <sub>2</sub> CH <sub>2</sub> , m. 152°. <sup>996</sup>   |
| (EtO) <sub>2</sub> CHCH <sub>2</sub> ,               | (EtO) <sub>2</sub> CHCH <sub>2</sub> , m. 54°. <sup>871</sup>   |
| MeSCH <sub>2</sub> CH <sub>2</sub> ,                 | MeSCH <sub>2</sub> CH <sub>2</sub> , disulfoxide, m. 141°; <sup>1143</sup> di-<br>sulfone, m. 220°. <sup>1143</sup> |
| EtSCH <sub>2</sub> CH <sub>2</sub> ,                 | EtSCH <sub>2</sub> CH <sub>2</sub> , m. 18°; <sup>1143</sup> disulfone, m.<br>141°. <sup>1143</sup>                 |
| Et <sub>2</sub> NCH <sub>2</sub> CH <sub>2</sub> ,   | Et <sub>2</sub> NCH <sub>2</sub> CH <sub>2</sub> , m. 86°. <sup>1062</sup>  |
| MeSCH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> , | MeSCH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> , m. 56°. <sup>1143</sup>  |
| CN,  | 2-Pyridyl, m. 203°. <sup>381</sup>  |
| CN,  | 6-Quinolyl, m. 181°. <sup>381</sup>   |

## R=Phenyl \*

| R'   |  |
|--|--|
| MeOCH <sub>2</sub> ,   | m. 133°. <sup>674</sup>                      |
| EtOCH <sub>2</sub> ,   | m. 126°; <sup>674</sup> 136°. <sup>551</sup> |
| <i>i</i> -AmOCH <sub>2</sub>   | m. 109°. <sup>674</sup>                      |
| 2,4-Cl <sub>2</sub> C <sub>6</sub> H <sub>3</sub> OCH <sub>2</sub> , | m. 85°. <sup>58</sup>                        |
| Me <sub>3</sub> SiCH <sub>2</sub> ,                                  | m. 123°. <sup>959</sup>                      |
| HOCH <sub>2</sub> CH <sub>2</sub> ,                                  | m. 61°. <sup>917</sup>                       |
| Me <sub>2</sub> NCH <sub>2</sub> CH <sub>2</sub> ,                   | m. 83°. <sup>177</sup>                       |
| Me <sub>2</sub> NCH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> ,   | m. 110°. <sup>177</sup>                      |
| Et <sub>2</sub> NCH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> ,   | m. 116°. <sup>177</sup>                      |
| Bu <sub>2</sub> NCH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> ,   | m. 93°. <sup>177</sup>                       |
| Piperidinopropyl,  | m. 93°. <sup>177</sup>                       |
| MeSO <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> ,  | m. 138°. <sup>732</sup>                      |
| MeSO <sub>2</sub> (CH <sub>2</sub> ) <sub>4</sub> ,                  | m. 105°. <sup>724</sup>                      |

\* For R'=alkyl see list for the alkyl.

| R  | R'  |
|--|---|
| Phenyl,  | m. 150.5°; <sup>524</sup> 152°; <sup>324a</sup> 153°; <sup>91</sup> 154°; <sup>254, 1389</sup><br>208°. <sup>947</sup>  |
| PhCH <sub>2</sub> ,  | m. 153.5°; <sup>374</sup> 154°. <sup>182</sup>  |
| <i>p</i> -HOC <sub>6</sub> H <sub>4</sub> CH <sub>2</sub> ,  | m. 168°. <sup>733</sup>   |
| <i>m</i> -MeOC <sub>6</sub> H <sub>4</sub> CH <sub>2</sub> ,   | m. 72.8°. <sup>375</sup>  |
| PhCHMe,  | m. 105°. <sup>182</sup>   |
| PhCH <sub>2</sub> CH <sub>2</sub> ,  | m. 111°. <sup>134</sup>   |
| PhCMe <sub>2</sub> CH <sub>2</sub> ,   | m. 106°. <sup>136</sup>   |
| PhCH <sub>2</sub> CH <sub>2</sub> CH,  | m. 77°. <sup>134</sup>  |
| ClC <sub>6</sub> H <sub>4</sub> , <i>o</i> , m.  | 163°; <sup>495</sup> 158°; <sup>1389</sup> 156°; <sup>978</sup> <i>m</i> , m. 116°; <sup>978</sup> <i>p</i> , m.<br>152°. <sup>978</sup>                      |
| 2,5-Cl <sub>2</sub> C <sub>6</sub> H <sub>3</sub> , m.   | 177°. <sup>172</sup>  |
| BrC <sub>6</sub> H <sub>4</sub> , <i>o</i> , m.  | 146°; <sup>978</sup> <i>m</i> , m. 97°; <sup>978</sup> <i>p</i> , m. 148°; <sup>978</sup> 151°. <sup>1094</sup>   |
| <i>p</i> -IC <sub>6</sub> H <sub>4</sub> , m.  | 153°. <sup>978</sup>  |
| <i>p</i> -FC <sub>6</sub> H <sub>4</sub> , m.  | 194°. <sup>172</sup>  |
| <i>p</i> -HOC <sub>6</sub> H <sub>4</sub> , m.   | 150°. <sup>978</sup>  |
| 3,4-(HO)(HOOC)C <sub>6</sub> H <sub>3</sub> , m.   | 172°; <sup>34</sup> 184°; <sup>1185</sup> Me ester, m. 140°; <sup>34</sup><br>Et ester, m. 128°. <sup>34</sup>  |
| <i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> , m.  | 143°. <sup>182</sup>  |
| 2,5-(MeO)(O <sub>2</sub> N)C <sub>6</sub> H <sub>3</sub> , m.  | 173°. <sup>341</sup>  |
| <i>p</i> -Et <sub>2</sub> NCH <sub>2</sub> CH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , HCl, m.            | 157°; <sup>626</sup> 163°. <sup>110</sup>   |
| <i>p</i> -BuOC <sub>6</sub> H <sub>4</sub> , m.  | 137°. <sup>583</sup>  |
| <i>p</i> -PhOC <sub>6</sub> H <sub>4</sub> , m.  | 140°; <sup>791</sup> 146°. <sup>173</sup>   |
| <i>o</i> -MeSC <sub>6</sub> H <sub>4</sub> , m.  | 189°. <sup>172</sup>  |
| <i>o</i> -MeSeC <sub>6</sub> H <sub>4</sub> , m.   | 196°. <sup>172</sup>  |
| O <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> , <i>o</i> , m.  | 188°; <sup>341</sup> <i>m</i> , m. 156°; <sup>341</sup> 157°; <sup>1095</sup> 158.5°; <sup>1243</sup> <i>p</i> ,<br>m. 175°. <sup>341</sup>                   |
| 3,4-(HOOC) <sub>2</sub> C <sub>6</sub> H <sub>3</sub> , m.   | 195°. <sup>384c</sup>   |
| H <sub>2</sub> NSO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> , <i>m</i> , m.                                  | 161.5°; <sup>1185</sup> <i>p</i> , m. 190°. <sup>1327</sup>   |
| <i>p</i> -(4-O <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> SO <sub>2</sub> )C <sub>6</sub> H <sub>4</sub> , m. | 180°. <sup>654</sup>  |
| <i>m</i> -(5-Chloro-2-pyrimidylsulfonyl)C <sub>6</sub> H <sub>4</sub> , m.                                     | 183°. <sup>759</sup>  |
| MeC <sub>6</sub> H <sub>4</sub> , <i>o</i> , m.  | 142°; <sup>947</sup> 138°; <sup>1389</sup> 136°; <sup>978</sup> <i>m</i> , m. 94°; <sup>978</sup> <i>p</i> , m.<br>140°; <sup>1389</sup> 141°. <sup>978</sup> |
| 2,4-BrMeC <sub>6</sub> H <sub>3</sub> , m.   | 154.5°. <sup>978</sup>  |
| 4,2-(HO)MeC <sub>6</sub> H <sub>3</sub> , m.   | 167.5°. <sup>978</sup>  |
| 2,5-(MeO)MeC <sub>6</sub> H <sub>3</sub> , m.  | 181°. <sup>172</sup>  |
| (O <sub>2</sub> N)MeC <sub>6</sub> H <sub>3</sub> , 5,2-, m.   | 171°; <sup>341</sup> 4,3-, m. 158°; 2,4-, m. 125°; <sup>341</sup><br>143°; <sup>1243</sup> 3,4-, m. 152°. <sup>341</sup>                                      |
| 2,4-Me <sub>2</sub> C <sub>6</sub> H <sub>3</sub> , m.   | 133.5°. <sup>978</sup>  |
| <i>p</i> -EtC <sub>6</sub> H <sub>4</sub> , m.   | 155°. <sup>172</sup>  |

## R

- $p$ -PrC<sub>6</sub>H<sub>4</sub>, m. 126°. <sup>172</sup>  
 $p$ -BuC<sub>6</sub>H<sub>4</sub>, m. 144°. <sup>172</sup>  
 $o$ -( $t$ -Bu)C<sub>6</sub>H<sub>4</sub>, m. 190°. <sup>172</sup>  
 $p$ -PhC<sub>6</sub>H<sub>4</sub>, m. 192°, <sup>141</sup> 208°. <sup>172</sup>  
C<sub>10</sub>H<sub>7</sub>,  $\alpha$  m. 159°, <sup>864</sup> 161°; <sup>996</sup>  $\beta$  m. 157°. <sup>864</sup>  
8-(5,6-Dimethylquinolyl), m. 158°. <sup>380</sup>  
2-Pyridyl, m. 167°, <sup>1082</sup> 168°, <sup>381</sup> 173°. <sup>1185</sup>  
3-Pyridyl, m. 163°. <sup>381</sup>  
5-Phenyl-2-oxazolyl, m. 195°. <sup>432</sup>  
2-Thiazolyl, m. 130°. <sup>432</sup>  
4-Methyl-2-thiazolyl, m. 174°. <sup>1185</sup>  
5-Methyl-2-thiazolyl, m. 172°. <sup>432</sup>  
5-Phenyl-2-thiazolyl, m. 150°. <sup>432</sup>  
2-Benzothiazolyl, m. 207°. <sup>698</sup>  
6-Chloro-2-benzothiazolyl, m. 174°. <sup>698</sup>  
5-Chloro-2-thenyl, m. 120°. <sup>534, 553</sup>  
5-Methyl-2-thenyl, m. 134°; <sup>534</sup> HCl, m. 197°. <sup>534</sup>  
5- $t$ -Butyl-2-thenyl, m. 186°. <sup>534</sup>  
5-Anilino-3-(1,2,4-triazolyl), m. 203°. <sup>432</sup>  
3-Benzylthio-1-phenyl-5-(1,2,4-triazolyl), m. 154°. <sup>432</sup>  
5-Benzylthio-1-phenyl-3-(1,2,4-triazolyl), m. 188°. <sup>432</sup>  
5-Methylthio-1-phenyl-3-(1,2,4-triazolyl), m. 178°. <sup>432</sup>  
1-Phenyl-5-thio-3-(1,2,4-triazolyl), m. 264°. <sup>432</sup>  
Menthyl, m. 179°. <sup>901</sup>  
Amidino, picrate, m. 224° dec. <sup>780b</sup>  
Methylamidino, m. 137°; <sup>780b</sup> HCl, m. 87°. <sup>780b</sup>  
Phenylamidino, m. 132°. <sup>780b</sup>  
Morpholino, m. 167°. <sup>1425</sup>  
Nicotinyl, m. 155°. <sup>320</sup>  
Xanthyl, m. 170° dec. <sup>415</sup>

R=PhCH<sub>2</sub>

## R'

- MeS(CH<sub>2</sub>)<sub>4</sub>, m. 78°. <sup>726</sup>  
PhCH<sub>2</sub>, m. 114°, <sup>1255</sup> 146°, <sup>947</sup> 148°. <sup>1105, 1304</sup>  
ClC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>,  $o$ , m. 165°; <sup>1167</sup>  $m$ , m. 84°; <sup>1167</sup>  $p$ , m. 140°. <sup>1167</sup>  
BrC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>,  $o$ , m. 100°; <sup>1167</sup>  $m$ , m. 90°; <sup>1167</sup>  $p$ , m. 139°. <sup>1167</sup>  
 $p$ -IC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>, m. 103°. <sup>1167</sup>  
 $p$ -MeOC<sub>6</sub>H<sub>4</sub>, m. 109°. <sup>182</sup>

R'

*p*-PhC<sub>6</sub>H<sub>4</sub>, m. 147°. <sup>150</sup>C<sub>10</sub>H<sub>7</sub>, α m. 172°; <sup>1262</sup> β m. 173°. <sup>150</sup>R=PhC<sub>2</sub>H<sub>4</sub>, PhC<sub>3</sub>H<sub>6</sub> and Others

| R   | R'  |  |
|---|---|--|
| PhCH <sub>2</sub> CH <sub>2</sub> ,                 | PhCH <sub>2</sub> CH <sub>2</sub> ,                 | m. 95°. <sup>134</sup>                       |
| PhCHMe,   | PhCHMe,   | m. 159°. <sup>841</sup>                      |
|   | <i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> ,        | m. 112.5°. <sup>182</sup>                    |
| PhCH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> , | PhCH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> , | m. 100°. <sup>134</sup>                      |
| PhCMe <sub>2</sub> ,                                | PhCMe <sub>2</sub> ,                                | m. 152°. <sup>841</sup>                      |
| Ph(CH <sub>2</sub> ) <sub>4</sub> ,                 | Ph(CH <sub>2</sub> ) <sub>4</sub> ,                 | m. 49°. <sup>134</sup>                       |
| Ph <sub>2</sub> CH,                                 | Ph <sub>2</sub> CH,                                 | m. 210°. <sup>272</sup> 211°. <sup>134</sup> |
| Ph <sub>3</sub> C,                                  | Ph <sub>3</sub> C,                                  | m. 210°. <sup>137</sup>                      |

R=*o*-ClC<sub>6</sub>H<sub>4</sub>

R'

*o*-ClC<sub>6</sub>H<sub>4</sub>, m. 128°. <sup>947</sup> 131.5°. <sup>435</sup>*p*-BrC<sub>6</sub>H<sub>4</sub>, m. 198°. <sup>172</sup>*p*-FC<sub>6</sub>H<sub>4</sub>, m. 189°. <sup>172</sup>3,4-(HO)(HOOC)C<sub>6</sub>H<sub>3</sub>, m. 196°. <sup>34</sup>*p*-MeOC<sub>6</sub>H<sub>4</sub>, m. 155°. <sup>182</sup> 173°. <sup>172</sup>*p*-EtOC<sub>6</sub>H<sub>4</sub>, m. 191°. <sup>172</sup>MeC<sub>6</sub>H<sub>4</sub>, *o*, m. 140°; <sup>978</sup> *p*, m. 184°. <sup>172</sup>*p*-PhC<sub>6</sub>H<sub>4</sub>, m. 197°. <sup>141</sup>R=*m*-ClC<sub>6</sub>H<sub>4</sub>

R'

*p*-FC<sub>6</sub>H<sub>4</sub>, m. 136°. <sup>172</sup>3,4-(HO)(HOOC)C<sub>6</sub>H<sub>3</sub>, m. 186°. <sup>34</sup>*p*-EtOC<sub>6</sub>H<sub>4</sub>, m. 133°. <sup>172</sup>*p*-H<sub>2</sub>NSO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>, m. 210°. <sup>1327</sup>MeC<sub>6</sub>H<sub>4</sub>, *o*, m. 124°; <sup>978</sup> *p*, m. 138°. <sup>172</sup>α-C<sub>10</sub>H<sub>7</sub>, m. 156°. <sup>172</sup>R=*p*-ClC<sub>6</sub>H<sub>4</sub>

R'

Amidino, m. 191° dec. <sup>101</sup>Morpholinopropyl, m. 102°. <sup>173</sup>*p*-ClC<sub>6</sub>H<sub>4</sub>, m. 166°. <sup>831</sup> 168°. <sup>76, 583</sup> 172°. <sup>753</sup>

R'

|   |   |
|---|---|
| 2,5-Cl <sub>2</sub> C <sub>6</sub> H <sub>3</sub> ,                           | m. 201°. <sup>172</sup>   |
| 2,3-Cl(NO <sub>2</sub> )C <sub>6</sub> H <sub>3</sub> ,                       | m. 151°. <sup>341</sup>   |
| <i>p</i> -BrC <sub>6</sub> H <sub>4</sub> ,                                   | m. 190°, <sup>1094</sup> 202°. <sup>172</sup>   |
| <i>p</i> -FC <sub>6</sub> H <sub>4</sub> ,                                    | m. 136°. <sup>172</sup>   |
| (F) (MeO)C <sub>6</sub> H <sub>3</sub> ,                                      | 2,5-, m. 161°; <sup>172</sup> 4,6-, m. 139°. <sup>172</sup>                                     |
| 3,4-(HO) (HOOC)C <sub>6</sub> H <sub>3</sub> ,                                | m. 198°. <sup>34</sup>  |
| <i>p</i> -EtOC <sub>6</sub> H <sub>4</sub> ,                                  | m. 189°. <sup>172</sup>   |
| <i>p</i> -PrOC <sub>6</sub> H <sub>4</sub> ,                                  | m. 172°, <sup>585</sup> 189°. <sup>172</sup>  |
| <i>p</i> - <i>i</i> -PrOC <sub>6</sub> H <sub>4</sub> ,                       | m. 172°. <sup>585</sup>   |
| <i>p</i> -BuOC <sub>6</sub> H <sub>4</sub> ,                                  | m. 168°. <sup>583, 585</sup>  |
| <i>p</i> -AmOC <sub>6</sub> H <sub>4</sub> ,                                  | m. 172°. <sup>172</sup>   |
| <i>p</i> - <i>i</i> -AmOC <sub>6</sub> H <sub>4</sub> ,                       | m. 170°, <sup>585</sup> 186°. <sup>172</sup>  |
| <i>p</i> -HexOC <sub>6</sub> H <sub>4</sub> ,                                 | m. 164°. <sup>585</sup>   |
| <i>p</i> -HepOC <sub>6</sub> H <sub>4</sub> ,                                 | m. 158°. <sup>585</sup>   |
| <i>p</i> -CH <sub>2</sub> :CHCH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , | m. 166°. <sup>585</sup>   |
| <i>p</i> -PhOC <sub>6</sub> H <sub>4</sub> ,                                  | m. 188°. <sup>173</sup>   |
| <i>o</i> -MeSC <sub>6</sub> H <sub>4</sub> ,                                  | m. 217°. <sup>172</sup>   |
| <i>o</i> -MeSeC <sub>6</sub> H <sub>4</sub> ,                                 | m. 226°. <sup>172</sup>   |
| O <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> ,                               | <i>m</i> , m. 170°; <sup>1095</sup> <i>p</i> , m. 179°, <sup>341</sup> 198°. <sup>446</sup>     |
| <i>p</i> -AcNHC <sub>6</sub> H <sub>4</sub> ,                                 | m. 202°. <sup>446</sup>   |
| <i>p</i> -H <sub>2</sub> NSO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> ,     | m. 180.5°. <sup>1327</sup>  |
| <i>m</i> -(5-Chloro-2-pyrimidylsulfonyl)C <sub>6</sub> H <sub>4</sub> ,       | m. 184° dec. <sup>759</sup>   |
| MeC <sub>6</sub> H <sub>4</sub> ,   | <i>o</i> -, m. 119.5°, <sup>201</sup> 134.5°; <sup>978</sup> <i>p</i> , m. 173°. <sup>201</sup> |
| 4,3-FMeC <sub>6</sub> H <sub>3</sub> ,  | m. 181°. <sup>172</sup>   |
| <i>p</i> -EtC <sub>6</sub> H <sub>4</sub> ,                                   | m. 197°. <sup>172</sup>   |
| <i>p</i> -PrC <sub>6</sub> H <sub>4</sub> ,                                   | m. 164.5°, <sup>585</sup> 177°. <sup>172</sup>  |
| <i>p</i> -BuC <sub>6</sub> H <sub>4</sub> ,                                   | m. 157°, <sup>585</sup> 174°. <sup>172</sup>  |
| <i>p</i> - <i>s</i> -BuC <sub>6</sub> H <sub>4</sub> ,                        | m. 164°. <sup>585</sup>   |
| <i>p</i> - <i>i</i> -AmC <sub>6</sub> H <sub>4</sub> ,                        | m. 164.5°. <sup>585</sup>   |
| <i>p</i> -HepC <sub>6</sub> H <sub>4</sub> ,                                  | m. 167°. <sup>172</sup>   |
| <i>p</i> -PhC <sub>6</sub> H <sub>4</sub> ,                                   | m. 191°, <sup>172</sup> 195°. <sup>141</sup>  |
| C <sub>10</sub> H <sub>7</sub> ,  | α m. 192°; <sup>172</sup> β m. 215°. <sup>172</sup>   |
| 2-Pyridyl,  | m. 188°, <sup>1018</sup> 208°. <sup>172</sup>   |
| 2-Thiazolyl,  | m. 204°. <sup>172</sup>   |

R=Cl<sub>2</sub>C<sub>6</sub>H<sub>3</sub> and Cl<sub>3</sub>C<sub>6</sub>H<sub>2</sub>

R

R'

|   |   |                          |
|---|---|--------------------------|
| 2,3-Cl <sub>2</sub> C <sub>6</sub> H <sub>3</sub> , | 2,3-Cl <sub>2</sub> C <sub>6</sub> H <sub>3</sub> , | m. 140°. <sup>343</sup>  |
| 2,4-Cl <sub>2</sub> C <sub>6</sub> H <sub>3</sub> , | 2,4-Cl <sub>2</sub> C <sub>6</sub> H <sub>3</sub> , | m. 185°. <sup>1033</sup> |
|   | <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> ,         | m. 145°. <sup>201</sup>  |

| R   | R'   |   |
|---|--|---|
| 2,5-Cl <sub>2</sub> C <sub>6</sub> H <sub>3</sub> ,   | 2,5-Cl <sub>2</sub> C <sub>6</sub> H <sub>3</sub> ,                          | m. 176°. <sup>1033</sup>  |
|   | <i>p</i> -BrC <sub>6</sub> H <sub>4</sub> ,                                  | m. 193°. <sup>172</sup>   |
|   | <i>p</i> -FC <sub>6</sub> H <sub>4</sub> ,                                   | m. 174°. <sup>172</sup>   |
|   | <i>o</i> -MeOC <sub>6</sub> H <sub>4</sub> ,                                 | m. 154°. <sup>172</sup>   |
|   | <i>p</i> -EtOC <sub>6</sub> H <sub>4</sub> ,                                 | m. 172°. <sup>172</sup>   |
|   | <i>p</i> -PrOC <sub>6</sub> H <sub>4</sub> ,                                 | m. 158°. <sup>172</sup>   |
|   | <i>p</i> -Me <sub>2</sub> CHOC <sub>6</sub> H <sub>4</sub> ,                 | m. 151°. <sup>172</sup>   |
|   | <i>p</i> -BuOC <sub>6</sub> H <sub>4</sub> ,                                 | m. 145°. <sup>172</sup>   |
|   | <i>p</i> -Me <sub>2</sub> CHCH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , | m. 161°. <sup>172</sup>   |
|   | MeC <sub>6</sub> H <sub>4</sub> ,  | <i>m</i> , m. 156°; <sup>172</sup> <i>p</i> , m. 162°. <sup>172</sup> |
|   | C <sub>10</sub> H <sub>7</sub> ,   | α m. 202°; <sup>172</sup> β m. 179°. <sup>172</sup>                   |
| 3,4-Cl <sub>2</sub> C <sub>6</sub> H <sub>3</sub> ,   | 3,4-Cl <sub>2</sub> C <sub>6</sub> H <sub>3</sub> ,                          | m. 181°. <sup>172</sup>   |
|   | α-C <sub>10</sub> H <sub>7</sub> ,   | m. 191°. <sup>172</sup>   |
| 3,5-Cl <sub>2</sub> C <sub>6</sub> H <sub>3</sub> ,   | 3,5-Cl <sub>2</sub> C <sub>6</sub> H <sub>3</sub> ,                          | m. 157°. <sup>1033</sup>  |
| 2,4,5-Cl <sub>3</sub> C <sub>6</sub> H <sub>2</sub> , | 2,4,5-Cl <sub>3</sub> C <sub>6</sub> H <sub>2</sub> ,                        | m. 162°. <sup>343</sup>   |
| 3,4,5-Cl <sub>3</sub> C <sub>6</sub> H <sub>2</sub> , | 3,4,5-Cl <sub>3</sub> C <sub>6</sub> H <sub>2</sub> ,                        | m. 140°. <sup>343</sup>   |

## R=Chlorophenyl Containing Other Substituents

| R  | R'   |                          |
|--|--|--------------------------|
| 2,4-ClBrC <sub>6</sub> H <sub>3</sub> ,      | 2,4-ClBrC <sub>6</sub> H <sub>3</sub> ,      | m. 160°. <sup>1033</sup> |
| 4,2-ClBrC <sub>6</sub> H <sub>3</sub> ,      | 4,2-ClBrC <sub>6</sub> H <sub>3</sub> ,      | m. 207°. <sup>1033</sup> |
| 3,4-(Cl)(MeO)C <sub>6</sub> H <sub>3</sub> , | α-C <sub>10</sub> H <sub>7</sub> ,           | m. 174°. <sup>153</sup>  |
| 4,2-(Cl)(MeO)C <sub>6</sub> H <sub>3</sub> , | 4,2-(Cl)(MeO)C <sub>6</sub> H <sub>3</sub> , | m. 152°. <sup>172</sup>  |
| 4,3-(Cl)(MeO)C <sub>6</sub> H <sub>3</sub> , | α-C <sub>10</sub> H <sub>7</sub> ,           | m. 155°. <sup>153</sup>  |
| 5,2-(Cl)(MeO)C <sub>6</sub> H <sub>3</sub> , | 5,2-(Cl)(MeO)C <sub>6</sub> H <sub>3</sub> , | m. 166°. <sup>368</sup>  |
| 5,3-(Cl)(MeO)C <sub>6</sub> H <sub>3</sub> , | <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> ,  | m. 136°. <sup>153</sup>  |

R=BrC<sub>6</sub>H<sub>4</sub> and Br<sub>2</sub>C<sub>6</sub>H<sub>3</sub>

| R   | R'   |   |
|---|--|---|
| <i>o</i> -BrC <sub>6</sub> H <sub>4</sub> , | <i>o</i> -BrC <sub>6</sub> H <sub>4</sub> ,  | m. 154°. <sup>435</sup> 157°. <sup>343</sup>                            |
|   | <i>o</i> -MeC <sub>6</sub> H <sub>4</sub> ,  | m. 128°. <sup>978</sup>   |
| <i>m</i> -BrC <sub>6</sub> H <sub>4</sub> , | <i>m</i> -BrC <sub>6</sub> H <sub>4</sub> ,  | m. 132°. <sup>435</sup>   |
|   | <i>o</i> -MeC <sub>6</sub> H <sub>4</sub> ,  | m. 101°. <sup>978</sup>   |
| <i>p</i> -BrC <sub>6</sub> H <sub>4</sub> , | <i>p</i> -BrC <sub>6</sub> H <sub>4</sub> ,  | m. 174°. <sup>585</sup> 180°. <sup>133</sup> 187°. <sup>1094</sup>      |
|   | <i>p</i> -FC <sub>6</sub> H <sub>4</sub> ,   | m. 177°. <sup>172</sup>   |
|   | 3,6-(F)(MeO)C <sub>6</sub> H <sub>3</sub> ,  | m. 152°. <sup>172</sup>   |
|   | HOC <sub>6</sub> H <sub>4</sub> ,            | <i>o</i> , m. 132°; <sup>1094</sup> <i>p</i> , m. 164°. <sup>1094</sup> |
|   |  | 196°. <sup>172</sup>  |
|   | <i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> , | m. 179°. <sup>182</sup> 202°. <sup>172</sup>                            |



| R   | R'  |
|---|---|
|   | 2,5-(MeO)(O <sub>2</sub> N)C <sub>6</sub> H <sub>3</sub> , m. 180°. <sup>341</sup>  |
|   | <i>p</i> -EtOC <sub>6</sub> H <sub>4</sub> , m. 179°. <sup>172</sup>  |
|   | <i>p</i> -PrOC <sub>6</sub> H <sub>4</sub> , m. 179°. <sup>585</sup>  |
|   | <i>p</i> -BuOC <sub>6</sub> H <sub>4</sub> , m. 171°, <sup>585</sup> 189°. <sup>172</sup>   |
|   | <i>p</i> -Me <sub>2</sub> CHCH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , m. 198°. <sup>172</sup>  |
|   | <i>p</i> -Me <sub>2</sub> CHCH <sub>2</sub> CH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , m. 179°, <sup>585</sup> 191°. <sup>171</sup>   |
|   | <i>p</i> -HepOC <sub>6</sub> H <sub>4</sub> , m. 184°. <sup>172</sup>   |
|   | <i>o</i> -MeSC <sub>6</sub> H <sub>4</sub> , m. 217°. <sup>172</sup>  |
|   | <i>o</i> -MeSeC <sub>6</sub> H <sub>4</sub> , m. 232°. <sup>172</sup>   |
|   | <i>p</i> -Me <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> , m. 208°. <sup>172</sup>  |
|   | O <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> , <i>o</i> , m. 61°, <sup>1094</sup> 210°; <sup>341</sup> <i>m</i> , m. 164°, <sup>1094</sup> 173°; <sup>341</sup> , <sup>1095</sup> <i>p</i> , m. 164°, <sup>341</sup> 165°. <sup>1094</sup> |
|   | H <sub>2</sub> NSO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> , <i>m</i> , m. 168.5°; <sup>1185</sup> <i>p</i> , m. 186°. <sup>1327</sup>   |
|   | <i>m</i> -(5-Chloro-2-pyrimidylsulfonyl)C <sub>6</sub> H <sub>4</sub> , m. 185°. <sup>759</sup>   |
|   | MeC <sub>6</sub> H <sub>4</sub> , <i>o</i> , m. 143°, <sup>978</sup> 148°; <sup>1094</sup> <i>m</i> , m. 148°; <sup>1094</sup> <i>p</i> , m. 175°, <sup>1094</sup> 182°. <sup>201</sup>   |
|   | 3,4-BrMeC <sub>6</sub> H <sub>3</sub> , m. 159°. <sup>1094</sup>  |
|   | (O <sub>2</sub> N)MeC <sub>6</sub> H <sub>3</sub> , 5-2, m. 183°; <sup>341</sup> 6,2-, m. 164°; <sup>341</sup> 4,3-, m. 147°; <sup>341</sup> 2,4-, m. 156°; <sup>341</sup> 3,4-, m. 173°. <sup>341</sup>                                    |
|   | 2,5-Me <sub>2</sub> C <sub>6</sub> H <sub>3</sub> , m. 137°. <sup>172</sup>   |
|   | <i>p</i> -EtC <sub>6</sub> H <sub>4</sub> , m. 198°. <sup>172</sup>   |
|   | <i>p</i> -PrC <sub>6</sub> H <sub>4</sub> , m. 187°. <sup>172</sup>   |
|   | <i>p</i> -BuC <sub>6</sub> H <sub>4</sub> , m. 181°. <sup>172</sup>   |
|   | <i>o</i> -Me <sub>3</sub> CC <sub>6</sub> H <sub>4</sub> , m. 187°. <sup>172</sup>  |
|   | <i>p</i> -Me <sub>2</sub> CHCH <sub>2</sub> CH <sub>2</sub> C <sub>6</sub> H <sub>4</sub> , m. 181°. <sup>585</sup>   |
|   | <i>p</i> -HepC <sub>6</sub> H <sub>4</sub> , m. 176°. <sup>172</sup>  |
|   | PhC <sub>6</sub> H <sub>4</sub> , <i>o</i> , m. 178°; <sup>172</sup> <i>p</i> , m. 196°, <sup>141</sup> 229°. <sup>172</sup>  |
|   | C <sub>10</sub> H <sub>7</sub> , α m. 166°, <sup>1094</sup> 226°; <sup>172</sup> β m. 176°, <sup>1094</sup> 212°. <sup>172</sup>  |
|   | Amidino, m. 188°; <sup>780b</sup> picrate, m. 250°. <sup>780b</sup>   |
|   | 2-Pyridyl, m. 156°. <sup>172</sup>  |
|   | 2-Thiazolyl, m. 206°. <sup>172</sup>  |
| 2,5-Br <sub>2</sub> C <sub>6</sub> H <sub>3</sub> , | 2,5-Br <sub>2</sub> C <sub>6</sub> H <sub>3</sub> , m. 154°. <sup>343</sup>   |
| 3,4-Br <sub>2</sub> C <sub>6</sub> H <sub>3</sub> , | 3,4-Br <sub>2</sub> C <sub>6</sub> H <sub>3</sub> , m. 103°. <sup>1033</sup>  |
| 3,5-Br <sub>2</sub> C <sub>6</sub> H <sub>3</sub> , | 3,5-Br <sub>2</sub> C <sub>6</sub> H <sub>3</sub> , m. 135°. <sup>1033</sup>  |



| R  | R'   |   |
|--|--|---|
| <i>o</i> -IC <sub>6</sub> H <sub>4</sub> , | <i>o</i> -IC <sub>6</sub> H <sub>4</sub> ,   | m. 164° dec. <sup>343</sup>   |
| <i>m</i> -IC <sub>6</sub> H <sub>4</sub> , | <i>m</i> -IC <sub>6</sub> H <sub>4</sub> ,   | m. 134°. <sup>589</sup>   |
| <i>p</i> -IC <sub>6</sub> H <sub>4</sub> , | <i>p</i> -IC <sub>6</sub> H <sub>4</sub> ,   | m. 189° dec. <sup>435</sup>   |
|  | <i>p</i> -PrOC <sub>6</sub> H <sub>4</sub> ,   | m. 178°. <sup>585</sup>   |
|  | <i>p</i> -BuOC <sub>6</sub> H <sub>4</sub> ,   | m. 176°. <sup>585</sup>   |
|  | <i>p</i> -Me <sub>2</sub> CHCH <sub>2</sub> CH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , | m. 185°. <sup>585</sup>   |
|  | <i>p</i> -CH <sub>2</sub> :CHCH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> ,                | m. 165°. <sup>585</sup>   |
|  | H <sub>2</sub> NSO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> ,                              | <i>m</i> , m. 186°; <sup>1185</sup> <i>p</i> , m. 184.5°. <sup>1327</sup> |
|  | <i>m</i> -(5-Chloro-2-pyrimidylsulfonyl)C <sub>6</sub> H <sub>4</sub> ,                      | m. 181°. <sup>759</sup>   |
|  | <i>o</i> -MeC <sub>6</sub> H <sub>4</sub> ,  | m. 150°. <sup>978</sup>   |
|  | <i>p</i> -PrC <sub>6</sub> H <sub>4</sub> ,  | m. 177°. <sup>585</sup>   |
|  | <i>p</i> -BuC <sub>6</sub> H <sub>4</sub> ,  | m. 170°. <sup>585</sup>   |
|  | <i>p</i> -Me <sub>2</sub> CHCH <sub>2</sub> CH <sub>2</sub> C <sub>6</sub> H <sub>4</sub> ,  | m. 180°. <sup>585</sup>   |

#### R=FC<sub>6</sub>H<sub>4</sub> and Substituted Fluorophenyl

| R  | R'   |   |
|--|--|---|
| <i>m</i> -FC <sub>6</sub> H <sub>4</sub> , | <i>m</i> -FC <sub>6</sub> H <sub>4</sub> ,   | m. 127°, <sup>589</sup> 144°. <sup>153</sup>                          |
| <i>p</i> -FC <sub>6</sub> H <sub>4</sub> , | <i>p</i> -FC <sub>6</sub> H <sub>4</sub> ,   | m. 145°, <sup>153, 589</sup> 208°. <sup>172</sup>                     |
|  | 2,5-(F)(MeO)C <sub>6</sub> H <sub>3</sub> ,  | m. 142°. <sup>172</sup>   |
|  | <i>p</i> -HOC <sub>6</sub> H <sub>4</sub> ,  | m. 179°. <sup>172</sup>   |
|  | MeOC <sub>6</sub> H <sub>4</sub> ,   | <i>o</i> , m. 121°; <sup>172</sup> <i>p</i> , m. 172°. <sup>172</sup> |
|  | 2,5-(MeO) <sub>2</sub> C <sub>6</sub> H <sub>4</sub> ,                                       | m. 161°. <sup>172</sup>   |
|  | EtOC <sub>6</sub> H <sub>4</sub> ,   | <i>o</i> , m. 134°; <sup>172</sup> <i>p</i> , m. 172°. <sup>172</sup> |
|  | <i>p</i> -PrOC <sub>6</sub> H <sub>4</sub> ,   | m. 178°. <sup>172</sup>   |
|  | <i>p</i> -BuOC <sub>6</sub> H <sub>4</sub> ,   | m. 156°, <sup>585</sup> 168°. <sup>172</sup>                          |
|  | <i>p</i> -Me <sub>2</sub> CHCH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> ,                 | m. 179°. <sup>172</sup>   |
|  | <i>p</i> -AmOC <sub>6</sub> H <sub>4</sub> ,   | m. 168°. <sup>172</sup>   |
|  | <i>p</i> -Me <sub>2</sub> CHCH <sub>2</sub> CH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , | m. 171°. <sup>172</sup>   |
|  | <i>p</i> -HepOC <sub>6</sub> H <sub>4</sub> ,  | m. 162°. <sup>172</sup>   |
|  | <i>p</i> -PhCH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , | m. 139°. <sup>172</sup>   |
|  | <i>p</i> -PhOC <sub>6</sub> H <sub>4</sub> ,   | m. 154°. <sup>173</sup>   |
|  | <i>o</i> -MeSC <sub>6</sub> H <sub>4</sub> ,   | m. 217°. <sup>172</sup>   |
|  | <i>o</i> -MeSeC <sub>6</sub> H <sub>4</sub> ,  | m. 221°. <sup>172</sup>   |
|  | <i>p</i> -Me <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> ,                                   | m. 194°. <sup>172</sup>   |
|  | MeC <sub>6</sub> H <sub>4</sub> ,  | <i>m</i> , m. 125°; <sup>172</sup> <i>p</i> , m. 179°. <sup>172</sup> |

| R   | R'  |
|---|---|
|   | 4,3-(O <sub>2</sub> N)MeC <sub>6</sub> H <sub>3</sub> , m. 154°. <sup>341</sup>                                     |
|   | 2,4-Me <sub>2</sub> C <sub>6</sub> H <sub>3</sub> , m. 131°. <sup>172</sup>   |
|   | <i>p</i> -EtC <sub>6</sub> H <sub>4</sub> , m. 168°. <sup>172</sup>   |
|   | <i>p</i> -PrC <sub>6</sub> H <sub>4</sub> , m. 157°. <sup>172</sup>   |
|   | <i>p</i> -BuC <sub>6</sub> H <sub>4</sub> , m. 162°. <sup>172</sup>   |
|   | <i>p</i> -HepC <sub>6</sub> H <sub>4</sub> , m. 158°. <sup>172</sup>  |
|   | PhC <sub>6</sub> H <sub>4</sub> , <i>o</i> , m. 163°; <sup>172</sup> <i>p</i> , m. 221°. <sup>172</sup>             |
|   | C <sub>10</sub> H <sub>7</sub> , $\alpha$ m. 187°; <sup>172</sup> $\beta$ m. 203°. <sup>172</sup>                   |
|   | 1-(4-ClC <sub>10</sub> H <sub>6</sub> ), m. 198°. <sup>172</sup>  |
|   | 2-Pyridyl, m. 178°. <sup>172</sup>  |
|   | 2-Thiazolyl, m. 181°. <sup>172</sup>  |
| 3,6-F(MeO)C <sub>6</sub> H <sub>3</sub> , | <i>p</i> -EtOC <sub>6</sub> H <sub>4</sub> , m. 163°. <sup>172</sup>  |
|   | <i>p</i> -Me <sub>2</sub> CHCH <sub>2</sub> CH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , m. 93°. <sup>172</sup> |
|   | <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> , m. 148°. <sup>172</sup>   |
|   | C <sub>10</sub> H <sub>7</sub> , $\alpha$ - m. 137°; <sup>172</sup> $\beta$ - m. 137°. <sup>172</sup>               |

## R=Hydroxyphenyl

| R   | R'   |
|---|--|
| <i>o</i> -HOC <sub>6</sub> H <sub>4</sub> , | <i>m</i> -O <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> , m. 168°. <sup>1095</sup>                                   |
| <i>m</i> -HOC <sub>6</sub> H <sub>4</sub> , | <i>m</i> -HOC <sub>6</sub> H <sub>4</sub> , dec. without melting. <sup>342</sup>                                     |
| <i>p</i> -HOC <sub>6</sub> H <sub>4</sub> , | <i>p</i> -HOC <sub>6</sub> H <sub>4</sub> , m. 225°. <sup>947</sup>  |
|   | <i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> , m. 198°. <sup>172</sup>   |
|   | <i>p</i> -EtOC <sub>6</sub> H <sub>4</sub> , m. 196°. <sup>172</sup>   |
|   | <i>p</i> -PrOC <sub>6</sub> H <sub>4</sub> , m. 198°. <sup>172</sup>   |
|   | <i>p</i> -BuOC <sub>6</sub> H <sub>4</sub> , m. 186°. <sup>172</sup>   |
|   | <i>p</i> -AmOC <sub>6</sub> H <sub>4</sub> , m. 172°. <sup>172</sup>   |
|   | <i>p</i> -Me <sub>2</sub> CHCH <sub>2</sub> CH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , m. 158°. <sup>172</sup> |
|   | <i>m</i> -O <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> , m. 152°, <sup>1243</sup> 155°. <sup>1095</sup>             |
|   | C <sub>10</sub> H <sub>7</sub> , $\alpha$ - m. 208°; <sup>172</sup> $\beta$ - m. 224°. <sup>172</sup>                |
|   | <i>o</i> -MeC <sub>6</sub> H <sub>4</sub> , m. 158°. <sup>978</sup>  |
|   | <i>o</i> -PhC <sub>6</sub> H <sub>4</sub> , m. 168°. <sup>172</sup>  |

## R=Methoxyphenyl

| R  | R'   |
|--|--|
| <i>o</i> -MeOC <sub>6</sub> H <sub>4</sub> , | MeOC <sub>6</sub> H <sub>4</sub> , <i>o</i> , m. 134°, <sup>343, 753</sup> 135°; <sup>947</sup> <i>p</i> , m. 161°. <sup>172</sup> |

| R  | R'  |
|--|---|
|  | <i>p</i> -EtOC <sub>6</sub> H <sub>4</sub> , m. 129°. <sup>172</sup>  |
|  | MeC <sub>6</sub> H <sub>4</sub> , <i>o</i> , m. 126°; <sup>978</sup> <i>m</i> , m. 149°; <sup>172</sup> <i>p</i> ,<br>m. 149°. <sup>172</sup>   |
|  | 4,5-Me <sub>2</sub> C <sub>6</sub> H <sub>3</sub> , m. 139°. <sup>172</sup>   |
|  | <i>p</i> -PrC <sub>6</sub> H <sub>4</sub> , m. 129°. <sup>172</sup>   |
|  | <i>p</i> -BuC <sub>6</sub> H <sub>4</sub> , m. 121°. <sup>172</sup>   |
|  | <i>p</i> -PhC <sub>6</sub> H <sub>4</sub> , m. 200°, <sup>141</sup> 223°. <sup>172</sup>  |
|  | 3,4-(HO)(HOOC)C <sub>6</sub> H <sub>3</sub> , m. 198°. <sup>34</sup>  |
| <i>m</i> -MeOC <sub>6</sub> H <sub>4</sub> , | <i>m</i> -MeOC <sub>6</sub> H <sub>4</sub> , m. 125°, <sup>589</sup> 126°. <sup>348</sup>   |
|  | 3,4-(HO)(HOOC)C <sub>6</sub> H <sub>3</sub> , m. 161°. <sup>34</sup>  |
| <i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> , | Morpholinoethyl, m. 140°. <sup>178</sup>  |
|  | Morpholinopropyl, m. 107°. <sup>173</sup>   |
|  | <i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> , m. 186.5°, <sup>342</sup> 187°, <sup>182</sup> 188°, <sup>947</sup><br>189°, <sup>588</sup> 192°. <sup>800</sup>                             |
|  | 2,5-(MeO) <sub>2</sub> C <sub>6</sub> H <sub>3</sub> , m. 149°. <sup>172</sup>  |
|  | <i>p</i> -EtOC <sub>6</sub> H <sub>4</sub> , m. 157°, <sup>172</sup> 170.5°. <sup>1302</sup>  |
|  | <i>p</i> -BuOC <sub>6</sub> H <sub>4</sub> , m. 164°. <sup>172</sup>  |
|  | <i>p</i> -Me <sub>2</sub> CHCH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , m. 161°. <sup>172</sup>  |
|  | <i>p</i> -Me <sub>2</sub> CHCH <sub>2</sub> CH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , m. 155°. <sup>172</sup>  |
|  | <i>p</i> -PhCH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , m. 154°. <sup>172</sup>  |
|  | <i>p</i> -PhOC <sub>6</sub> H <sub>4</sub> , m. 169°. <sup>178</sup>  |
|  | <i>p</i> -O <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> , m. 155°. <sup>341</sup>   |
|  | 3,4-(HO)(HOOC)C <sub>6</sub> H <sub>3</sub> , <i>p</i> , m. 192°. <sup>34</sup>   |
|  | <i>p</i> -H <sub>2</sub> NSO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> , m. 176°. <sup>1327</sup>  |
|  | <i>m</i> -(5-Chloro-2-pyrimidylsulfonyl)C <sub>6</sub> H <sub>4</sub> , m.<br>170°. <sup>759</sup>  |
|  | <i>p</i> -Sulfamoyl C <sub>6</sub> H <sub>4</sub> , m. 234°. <sup>172</sup>   |
|  | MeC <sub>6</sub> H <sub>4</sub> , <i>o</i> , m. 138°, <sup>978</sup> 139°; <sup>182</sup> <i>m</i> , m. 140°, <sup>182</sup> <i>p</i> ,<br>m. 148.5°, <sup>182</sup> 161°. <sup>172</sup> |
|  | 2,3-Me <sub>2</sub> C <sub>6</sub> H <sub>3</sub> , m. 183°. <sup>172</sup>   |
|  | <i>p</i> -EtC <sub>6</sub> H <sub>4</sub> , m. 163°. <sup>172</sup>   |
|  | <i>p</i> -PrC <sub>6</sub> H <sub>4</sub> , m. 164°. <sup>172</sup>   |
|  | <i>p</i> -BuC <sub>6</sub> H <sub>4</sub> , m. 169°. <sup>172</sup>   |
|  | <i>p</i> -PhC <sub>6</sub> H <sub>4</sub> , m. 194°, <sup>182</sup> 211°. <sup>172</sup>  |
|  | C <sub>10</sub> H <sub>7</sub> , α- m. 163°, <sup>182</sup> 194°; <sup>172</sup> β m. 160°, <sup>182</sup><br>192°. <sup>172</sup>  |
|  | 2-Pyridyl, m. 208°. <sup>172</sup>  |
|  | 2-Thiazolyl, m. 212°. <sup>172</sup>  |

| R  | R'   |
|--|--|
| 2,5-(MeO) <sub>2</sub> C <sub>6</sub> H <sub>3</sub> ,     | 2,5-(MeO) <sub>2</sub> C <sub>6</sub> H <sub>3</sub> , m. 127°. <sup>343</sup>   |
|  | <i>p</i> -EtOC <sub>6</sub> H <sub>4</sub> , m. 149°. <sup>172</sup>   |
|  | C <sub>10</sub> H <sub>7</sub> , α m. 153°; <sup>172</sup> β- m. 190°. <sup>172</sup>  |
| 3,4-(MeO) <sub>2</sub> C <sub>6</sub> H <sub>3</sub> ,     | 3,4-(MeO) <sub>2</sub> C <sub>6</sub> H <sub>3</sub> , m. 140°. <sup>343</sup>   |
| 3,5-(MeO) <sub>2</sub> C <sub>6</sub> H <sub>3</sub> ,     | <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> , m. 148°. <sup>153</sup>  |
| 5,2-(O <sub>2</sub> N)(MeO)C <sub>6</sub> H <sub>3</sub> , | 5,2-(O <sub>2</sub> N)(MeO)C <sub>6</sub> H <sub>3</sub> , m. 175°. <sup>341</sup>   |
|  | O <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> , <i>m</i> , m. 164°; <sup>341</sup> <i>p</i> , m. 162°. <sup>341</sup>                      |
|  | MeC <sub>6</sub> H <sub>4</sub> , <i>o</i> , m. 162°; <sup>341</sup> <i>m</i> , m. 168°; <sup>182</sup> <i>p</i> , m. 162°. <sup>341</sup> |
|  | C <sub>10</sub> H <sub>7</sub> , α m. 178°; <sup>341</sup> β m. 175°. <sup>341</sup>   |

## R=Ethoxyphenyl

| R  | R'  |
|--|---|
| <i>o</i> -EtOC <sub>6</sub> H <sub>4</sub> , | EtOC <sub>6</sub> H <sub>4</sub> , <i>o</i> , m. 125°; <sup>343</sup> 126°; <sup>172</sup> <i>p</i> , m. 145°. <sup>583</sup> |
|  | <i>o</i> -MeC <sub>6</sub> H <sub>4</sub> , m. 156°. <sup>172</sup>   |
|  | <i>p</i> -EtC <sub>6</sub> H <sub>4</sub> , m. 148°. <sup>172</sup>   |
|  | <i>p</i> -PrC <sub>6</sub> H <sub>4</sub> , m. 161°. <sup>172</sup>   |
|  | <i>p</i> -BuC <sub>6</sub> H <sub>4</sub> , m. 138°. <sup>172</sup>   |
|  | <i>o</i> -Me <sub>3</sub> CC <sub>6</sub> H <sub>4</sub> , m. 172°. <sup>172</sup>  |
|  | <i>p</i> -PhC <sub>6</sub> H <sub>4</sub> , m. 232°. <sup>172</sup>   |
|  | α-C <sub>10</sub> H <sub>7</sub> , m. 164°. <sup>172</sup>  |
| <i>m</i> -EtOC <sub>6</sub> H <sub>4</sub> , | EtOC <sub>6</sub> H <sub>4</sub> , <i>m</i> , m. 115°; <sup>343</sup> <i>p</i> , m. 112°. <sup>583</sup>                      |
| <i>p</i> -EtOC <sub>6</sub> H <sub>4</sub> , | Morpholinoethyl, m. 136°. <sup>173</sup>  |
|  | Morpholinopropyl, m. 83°. <sup>173</sup>  |
|  | <i>p</i> -EtOC <sub>6</sub> H <sub>4</sub> , m. 170°; <sup>300</sup> 171°; <sup>342, 583</sup> 174°. <sup>1302</sup>          |
|  | <i>p</i> -HOCH <sub>2</sub> CH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , m. 174°. <sup>584</sup>                          |
|  | <i>p</i> -EtOCH <sub>2</sub> CH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , m. 146°. <sup>584</sup>                         |
|  | <i>p</i> -EtSCH <sub>2</sub> CH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , m. 140°. <sup>584</sup>                         |
|  | <i>p</i> -PrOC <sub>6</sub> H <sub>4</sub> , m. 187°. <sup>171</sup>  |
|  | <i>p</i> -BuOC <sub>6</sub> H <sub>4</sub> , m. 165.5°; <sup>1302</sup> 169°. <sup>171</sup>                                  |
|  | <i>p</i> -Me <sub>2</sub> CHCH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , m. 179°. <sup>171</sup>                          |
|  | <i>p</i> -AmOC <sub>6</sub> H <sub>4</sub> , m. 162°. <sup>171</sup>  |
|  | <i>p</i> -Me <sub>2</sub> CHCH <sub>2</sub> CH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , m. 172°. <sup>171</sup>          |
|  | <i>p</i> -HexOC <sub>6</sub> H <sub>4</sub> , m. 154°. <sup>583</sup>   |
|  | <i>p</i> -PhCH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , m. 147°. <sup>172</sup>          |
|  | <i>p</i> -PhOC <sub>6</sub> H <sub>4</sub> , m. 161°. <sup>173</sup>  |
|  | <i>o</i> -MeSC <sub>6</sub> H <sub>4</sub> , m. 202°. <sup>172</sup>  |
|  | <i>p</i> -EtSC <sub>6</sub> H <sub>4</sub> , m. 172°. <sup>584</sup>  |
|  | <i>p</i> -PrSC <sub>6</sub> H <sub>4</sub> , m. 160°. <sup>584</sup>  |

| R | R'   |
|---|--|
|   | <i>p</i> -Me <sub>2</sub> CHSC <sub>6</sub> H <sub>4</sub> , m. 149°. <sup>584</sup>   |
|   | <i>p</i> -BuSC <sub>6</sub> H <sub>4</sub> , m. 156°. <sup>584</sup>   |
|   | <i>p</i> -AmSC <sub>6</sub> H <sub>4</sub> , m. 156°. <sup>584</sup>   |
|   | <i>o</i> -MeSeC <sub>6</sub> H <sub>4</sub> , m. 207°. <sup>172</sup>  |
|   | <i>p</i> -Me <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> , m. 176°, <sup>585</sup> 197°. <sup>172</sup>                        |
|   | <i>p</i> -Sulfamoyl C <sub>6</sub> H <sub>4</sub> , m. 246°. <sup>172</sup>  |
|   | <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> , m. 142°. <sup>172</sup>  |
|   | <i>p</i> -EtOCH <sub>2</sub> C <sub>6</sub> H <sub>4</sub> , m. 136°. <sup>585</sup>   |
|   | 3,4-Me <sub>2</sub> C <sub>6</sub> H <sub>3</sub> , m. 122°. <sup>172</sup>  |
|   | <i>p</i> -EtC <sub>6</sub> H <sub>4</sub> , m. 158°. <sup>172</sup>  |
|   | <i>p</i> -MeOCH <sub>2</sub> CH <sub>2</sub> C <sub>6</sub> H <sub>4</sub> , m. 133°. <sup>585</sup>                           |
|   | <i>p</i> -PrC <sub>6</sub> H <sub>4</sub> , m. 166°. <sup>172</sup>  |
|   | <i>p</i> -MeOCH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> C <sub>6</sub> H <sub>4</sub> , m. 126°. <sup>585</sup>           |
|   | <i>p</i> -Me <sub>2</sub> CHC <sub>6</sub> H <sub>4</sub> , m. 139°. <sup>585</sup>  |
|   | <i>p</i> -BuC <sub>6</sub> H <sub>4</sub> , m. 149°, <sup>585</sup> 159°. <sup>172</sup>                                       |
|   | <i>p</i> -Me <sub>3</sub> CC <sub>6</sub> H <sub>4</sub> , m. 147°. <sup>585</sup>   |
|   | <i>p</i> -AmC <sub>6</sub> H <sub>4</sub> , m. 145°. <sup>585</sup>  |
|   | <i>p</i> -Me <sub>2</sub> CHCH <sub>2</sub> CH <sub>2</sub> C <sub>6</sub> H <sub>4</sub> , m. 142°. <sup>585</sup>            |
|   | <i>p</i> -Hex C <sub>6</sub> H <sub>4</sub> , m. 144°. <sup>585</sup>  |
|   | <i>p</i> -Hep C <sub>6</sub> H <sub>4</sub> , m. 151°. <sup>172</sup>  |
|   | PhC <sub>6</sub> H <sub>4</sub> , <i>o</i> , m. 144°. <sup>172</sup> ; <i>p</i> , m. 198°, <sup>141</sup> 226°. <sup>172</sup> |
|   | C <sub>10</sub> H <sub>7</sub> , $\alpha$ m. 201°. <sup>172</sup> ; $\beta$ m. 190°. <sup>172</sup>                            |
|   | 2-Pyridyl, m. 209°. <sup>172</sup>   |
|   | 2-Thiazolyl, m. 203°. <sup>172</sup>   |

R=Substituted Ethoxyphenyl

| R   | R' |
|---|----|
| <i>p</i> -HOCH <sub>2</sub> CH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , <i>p</i> -HOCH <sub>2</sub> CH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , m. 188°. <sup>584</sup>     |    |
| <i>p</i> -EtOCH <sub>2</sub> CH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , <i>p</i> -EtOCH <sub>2</sub> CH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , m. 137.5°. <sup>584</sup> |    |
| <i>p</i> -Me <sub>2</sub> NCH <sub>2</sub> CH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , <i>p</i> -PrOC <sub>6</sub> H <sub>4</sub> , m. 140°. <sup>585</sup>                      |    |
| <i>p</i> -BuOC <sub>6</sub> H <sub>4</sub> , m. 144°. <sup>585</sup>  |    |
| <i>p</i> -Me <sub>2</sub> CHCH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , HCl, m. 192°. <sup>585</sup>   |    |
| <i>p</i> -AmOC <sub>6</sub> H <sub>4</sub> , HCl, m. 167°. <sup>585</sup>   |    |
| <i>p</i> -Me <sub>2</sub> CHCH <sub>2</sub> CH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , m. 184°. <sup>585</sup>  |    |
| <i>p</i> -CH <sub>2</sub> :CHCH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , m. 150°. <sup>585</sup>   |    |
| <i>p</i> -PrC <sub>6</sub> H <sub>4</sub> , m. 140°. <sup>585</sup>   |    |
| <i>p</i> -Me <sub>2</sub> CHCH <sub>2</sub> C <sub>6</sub> H <sub>4</sub> , HCl, m. 144°. <sup>585</sup>  |    |
| <i>p</i> -AmC <sub>6</sub> H <sub>4</sub> , m. 167°. <sup>585</sup>   |    |
| <i>p</i> -Me <sub>2</sub> CHCH <sub>2</sub> CH <sub>2</sub> C <sub>6</sub> H <sub>4</sub> , HCl, m. 184°. <sup>585</sup>  |    |
| <i>p</i> -CH <sub>2</sub> :CHCH <sub>2</sub> C <sub>6</sub> H <sub>4</sub> , m. 150°. <sup>585</sup>  |    |

R

$p$ -Et<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>OC<sub>6</sub>H<sub>4</sub>,  $p$ -BuOC<sub>6</sub>H<sub>4</sub>, HCl, m. 156°. <sup>585</sup>  
 $p$ -Me<sub>2</sub>CHCH<sub>2</sub>OC<sub>6</sub>H<sub>4</sub>, HCl, m. 166°. <sup>585</sup>  
 $p$ -BuC<sub>6</sub>H<sub>4</sub>, HCl, m. 156°. <sup>585</sup>  
 $p$ -Me<sub>2</sub>CHCH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>, m. 86°; <sup>585</sup> HCl, m. 166°. <sup>585</sup>



R

 $p$ -PrOC<sub>6</sub>H<sub>4</sub>,

R'

$p$ -PrOC<sub>6</sub>H<sub>4</sub>, m. 182°, <sup>214b</sup> 186°. <sup>171</sup>  
 $p$ -BuOC<sub>6</sub>H<sub>4</sub>, m. 180°. <sup>171</sup>  
 $p$ -Me<sub>2</sub>CHCH<sub>2</sub>OC<sub>6</sub>H<sub>4</sub>, m. 175°. <sup>172</sup>  
 $p$ -AmOC<sub>6</sub>H<sub>4</sub>, m. 167°. <sup>172</sup>  
 $p$ -Me<sub>2</sub>CHCH<sub>2</sub>CH<sub>2</sub>OC<sub>6</sub>H<sub>4</sub>, m. 156°. <sup>171</sup>  
 $p$ -HepOC<sub>6</sub>H<sub>4</sub>, m. 162°. <sup>172</sup>  
 $p$ -PhCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>OC<sub>6</sub>H<sub>4</sub>, m. 143°. <sup>172</sup>  
 $o$ -MeSC<sub>6</sub>H<sub>4</sub>, m. 204°. <sup>172</sup>  
 $o$ -MeSeC<sub>6</sub>H<sub>4</sub>, m. 203°. <sup>172</sup>  
 $p$ -Me<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>, m. 149°, <sup>585</sup> 156°. <sup>172</sup>  
 $p$ -MeC<sub>6</sub>H<sub>4</sub>, m. 142°. <sup>172</sup>  
 $p$ -EtC<sub>6</sub>H<sub>4</sub>, m. 154°. <sup>172</sup>  
 $p$ -MeOCH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>, m. 137°. <sup>585</sup>  
 $p$ -PrC<sub>6</sub>H<sub>4</sub>, m. 150°, <sup>585</sup> 159°. <sup>172</sup>  
 $p$ -BuC<sub>6</sub>H<sub>4</sub>, m. 167°. <sup>172</sup>  
 $p$ -Me<sub>2</sub>CHCH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>, m. 133°. <sup>585</sup>  
 $p$ -HexC<sub>6</sub>H<sub>4</sub>, m. 145°. <sup>585</sup>  
 $p$ -HepC<sub>6</sub>H<sub>4</sub>, m. 156°. <sup>172</sup>  
2-Pyridyl, m. 171°. <sup>172</sup>

2,5-(PrO)(O<sub>2</sub>N)C<sub>6</sub>H<sub>3</sub>, 2,5-(PrO)(O<sub>2</sub>N)C<sub>6</sub>H<sub>3</sub>, m. 196.5°. <sup>1026</sup>

$p$ -Me<sub>2</sub>CHOC<sub>6</sub>H<sub>4</sub>,  $p$ -Me<sub>2</sub>CHOC<sub>6</sub>H<sub>4</sub>, m. 142°, <sup>172</sup> 144°. <sup>1302</sup>

$o$ -MeSC<sub>6</sub>H<sub>4</sub>, m. 144°. <sup>172</sup>  
 $o$ -MeSeC<sub>6</sub>H<sub>4</sub>, m. 151°. <sup>172</sup>  
 $p$ -EtC<sub>6</sub>H<sub>4</sub>, m. 134°. <sup>172</sup>  
 $p$ -BuC<sub>6</sub>H<sub>4</sub>, m. 138°. <sup>172</sup>  
 $p$ -HepC<sub>6</sub>H<sub>4</sub>, m. 151°. <sup>172</sup>



R

 $p$ -BuOC<sub>6</sub>H<sub>4</sub>,

R'

$p$ -BuOC<sub>6</sub>H<sub>4</sub>, m. 167°. <sup>583</sup>  
 $p$ -Me<sub>2</sub>CHCH<sub>2</sub>OC<sub>6</sub>H<sub>4</sub>, m. 162°. <sup>171</sup>  
 $p$ -AmOC<sub>6</sub>H<sub>4</sub>, m. 175°. <sup>171</sup>

| R   | R'   |
|---|--|
|   | <i>p</i> -Me <sub>2</sub> CHCH <sub>2</sub> CH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , m. 154°. <sup>171</sup> |
|   | <i>p</i> -PhCH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , m. 149°. <sup>172</sup> |
|   | <i>o</i> -MeSC <sub>6</sub> H <sub>4</sub> , m. 194°. <sup>172</sup>   |
|   | <i>o</i> -MeSeC <sub>6</sub> H <sub>4</sub> , m. 195°. <sup>172</sup>  |
|   | <i>p</i> -Me <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> , m. 121°. <sup>583, 585</sup>                              |
|   | <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> , m. 139°. <sup>172</sup>  |
|   | <i>p</i> -F <sub>3</sub> CC <sub>6</sub> H <sub>4</sub> , m. 186°. <sup>172</sup>                                    |
|   | <i>p</i> -MeOCH <sub>2</sub> CH <sub>2</sub> C <sub>6</sub> H <sub>4</sub> , m. 135°. <sup>585</sup>                 |
|   | <i>p</i> -PrC <sub>6</sub> H <sub>4</sub> , m. 157°. <sup>172</sup>  |
|   | <i>p</i> -BuC <sub>6</sub> H <sub>4</sub> , m. 152°. <sup>585</sup> 161°. <sup>172</sup>                             |
|   | <i>p</i> -HexC <sub>6</sub> H <sub>4</sub> , m. 144°. <sup>585</sup>   |
|   | <i>p</i> -HepC <sub>6</sub> H <sub>4</sub> , m. 151°. <sup>172</sup>   |
|   | C <sub>10</sub> H <sub>7</sub> , α m. 157°; <sup>172</sup> β m. 185°. <sup>172</sup>                                 |
|   | 1-(5-HOC <sub>10</sub> H <sub>8</sub> ), m. 181°. <sup>172</sup>   |
|   | 2-Pyridyl, m. 149°. <sup>172</sup>   |
| <i>p</i> -Me <sub>2</sub> CHCH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , <i>p</i> -Me <sub>2</sub> CHCH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , m. 148°. <sup>171</sup> |  |
|   | <i>p</i> -AmOC <sub>6</sub> H <sub>4</sub> , m. 143°. <sup>172</sup>   |
|   | <i>p</i> -Me <sub>2</sub> CHCH <sub>2</sub> CH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , m. 140°. <sup>171</sup> |
|   | <i>o</i> -MeSC <sub>6</sub> H <sub>4</sub> , m. 207°. <sup>172</sup>   |
|   | <i>o</i> -MeSeC <sub>6</sub> H <sub>4</sub> , m. 198°. <sup>172</sup>  |
|   | <i>p</i> -Me <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> , m. 147°. <sup>585</sup>                                   |
|   | <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> , m. 152°. <sup>172</sup>  |
|   | <i>p</i> -EtC <sub>6</sub> H <sub>4</sub> , m. 146°. <sup>172</sup>  |
|   | <i>p</i> -MeOCH <sub>2</sub> CH <sub>2</sub> C <sub>6</sub> H <sub>4</sub> , m. 125°. <sup>585</sup>                 |
|   | <i>p</i> -PrC <sub>6</sub> H <sub>4</sub> , m. 159°. <sup>172</sup>  |
|   | <i>p</i> -BuC <sub>6</sub> H <sub>4</sub> , m. 151°. <sup>172</sup>  |
|   | <i>p</i> -HepC <sub>6</sub> H <sub>4</sub> , m. 116°. <sup>172</sup>   |
|   | PhC <sub>6</sub> H <sub>4</sub> , <i>o</i> , m. 145°; <sup>172</sup> <i>p</i> , m. 225°. <sup>172</sup>              |
|   | C <sub>10</sub> H <sub>7</sub> , α m. 166°; <sup>172</sup> β m. 179°. <sup>172</sup>                                 |

R=Higher Alkoxyphenyl

| R   | R'   |
|---|--|
| <i>p</i> -AmOC <sub>6</sub> H <sub>4</sub> , <i>p</i> -AmOC <sub>6</sub> H <sub>4</sub> , m. 154°. <sup>171</sup>   |  |
|   | <i>p</i> -Me <sub>2</sub> CHCH <sub>2</sub> CH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , m. 139°. <sup>171</sup> |
|   | <i>p</i> -Me <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> , m. 142°. <sup>172</sup>                                   |
| <i>p</i> -Me <sub>2</sub> CHCH <sub>2</sub> CH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , <i>p</i> -Me <sub>2</sub> CHCH <sub>2</sub> CH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , m. 149°. <sup>171</sup> |  |
|   | <i>p</i> -HepOC <sub>6</sub> H <sub>4</sub> , m. 121°. <sup>172</sup>  |
|   | <i>p</i> -PhCH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , m. 132°. <sup>172</sup> |
|   | <i>p</i> -PhOC <sub>6</sub> H <sub>4</sub> , m. 135°. <sup>173</sup>   |



| R   | R'  |                      |
|---|---|----------------------|
|   | <i>o</i> -MeSC <sub>6</sub> H <sub>4</sub> ,  | m. 189°.172          |
|   | <i>o</i> -MeSeC <sub>6</sub> H <sub>4</sub> ,   | m. 189°.172          |
|   | <i>p</i> -Me <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> ,                                  | m. 142°.585 143°.171 |
|   | <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> ,   | m. 135°.172          |
|   | <i>p</i> -PrC <sub>6</sub> H <sub>4</sub> ,   | m. 132°.585 138°.172 |
|   | <i>p</i> -Me <sub>2</sub> CHC <sub>6</sub> H <sub>4</sub> ,                                 | m. 134°.585          |
|   | <i>p</i> -BuC <sub>6</sub> H <sub>4</sub> ,   | m. 132°.172          |
|   | <i>p</i> -Me <sub>2</sub> CHCH <sub>2</sub> CH <sub>2</sub> C <sub>6</sub> H <sub>4</sub> , | m. 142°.585          |
|   | <i>p</i> -HepC <sub>6</sub> H <sub>4</sub> ,  | m. 112°.172          |
|   | <i>p</i> -PhC <sub>6</sub> H <sub>4</sub> ,   | m. 195°.172          |
|   | $\alpha$ -C <sub>10</sub> H <sub>7</sub> ,  | m. 163°.172          |
|   | 2-Pyridyl,  | m. 146°.172          |
| <i>p</i> -HepOC <sub>6</sub> H <sub>4</sub> , | <i>p</i> -HepOC <sub>6</sub> H <sub>4</sub> ,   | m. 155°.172          |
|   | 2,4-Me <sub>2</sub> C <sub>6</sub> H <sub>3</sub> ,   | m. 124°.172          |
|   | <i>o</i> -PhC <sub>6</sub> H <sub>4</sub> ,   | m. 123°.172          |
|   | $\alpha$ -C <sub>10</sub> H <sub>7</sub> ,  | m. 176°.172          |
| <i>p</i> -OctOC <sub>6</sub> H <sub>4</sub> , | <i>p</i> -OctOC <sub>6</sub> H <sub>4</sub> ,   | m. 156°.583          |

R=PhOC<sub>6</sub>H<sub>4</sub> and PhCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>OC<sub>6</sub>H<sub>4</sub>

| R  | R'   |                                 |
|--|--|---------------------------------|
| <i>p</i> -PhOC <sub>6</sub> H <sub>4</sub> ,   | <i>p</i> -PhOC <sub>6</sub> H <sub>4</sub> ,   | m. 172°.719                     |
|  | <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> ,  | m. 161°.173                     |
|  | 2,4-Me <sub>2</sub> C <sub>6</sub> H <sub>3</sub> ,  | m. 154°.173                     |
|  | C <sub>10</sub> H <sub>7</sub> , $\alpha$  | m. 181°.173 $\beta$ m. 173°.173 |
| <i>p</i> -PhCH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , | <i>p</i> -PhCH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> , | m. 160°.172                     |
|  | <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> ,  | m. 130°.172                     |
|  | <i>m</i> -PhC <sub>6</sub> H <sub>4</sub> ,  | m. 131°.172                     |
|  | $\beta$ -C <sub>10</sub> H <sub>7</sub> ,  | m. 171°.172                     |

R=Alkylmercaptophenyl

| R  | R'  |             |
|--|---|-------------|
| <i>o</i> -MeSC <sub>6</sub> H <sub>4</sub> , | <i>o</i> -MeSC <sub>6</sub> H <sub>4</sub> ,  | m. 159°.172 |
|  | <i>m</i> -MeC <sub>6</sub> H <sub>4</sub> ,   | m. 174°.172 |
|  | <i>p</i> -PrC <sub>6</sub> H <sub>4</sub> ,   | m. 183°.172 |
|  | <i>p</i> -BuC <sub>6</sub> H <sub>4</sub> ,   | m. 179°.172 |
|  | $\beta$ -C <sub>10</sub> H <sub>7</sub> ,   | m. 205°.172 |
| <i>p</i> -EtSC <sub>6</sub> H <sub>4</sub> , | <i>p</i> -Me <sub>2</sub> CHCH <sub>2</sub> CH <sub>2</sub> C <sub>6</sub> H <sub>4</sub> , | m. 174°.585 |
| <i>p</i> -PrSC <sub>6</sub> H <sub>4</sub> , | <i>p</i> -PrSC <sub>6</sub> H <sub>4</sub> ,  | m. 161°.585 |
| <i>p</i> -BuSC <sub>6</sub> H <sub>4</sub> , | <i>p</i> -BuSC <sub>6</sub> H <sub>4</sub> ,  | m. 156°.585 |
| <i>p</i> -AmSC <sub>6</sub> H <sub>4</sub> , | <i>p</i> -AmC <sub>6</sub> H <sub>4</sub> ,   | m. 164°.585 |

## R=Alkylselenophenyl

| R   | R'  |  |
|---|---|--|
| <i>o</i> -MeSeC <sub>6</sub> H <sub>4</sub> , | <i>o</i> -MeSeC <sub>6</sub> H <sub>4</sub> , | m. 167°. <sup>172</sup>  |
|   | MeC <sub>6</sub> H <sub>4</sub> ,             | <i>m</i> , m. 176°. <sup>172</sup> ; <sup>172</sup> <i>p</i> , m. 209°. <sup>172</sup> |
|   | <i>p</i> -PrC <sub>6</sub> H <sub>4</sub> ,   | m. 182°. <sup>172</sup>  |
|   | <i>p</i> -BuC <sub>6</sub> H <sub>4</sub> ,   | m. 178°. <sup>172</sup>  |
|   | β-C <sub>10</sub> H <sub>7</sub> ,            | m. 210°. <sup>172</sup>  |

## R=Aminophenyl

| R  | R'   |   |
|--|--|---|
| <i>m</i> -Me <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> , | <i>m</i> -Me <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> , | m. 162°. <sup>589</sup>                             |
| <i>p</i> -Me <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> , | <i>p</i> -Me <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> , | m. 186°. <sup>583</sup> ; 187°. <sup>561</sup>      |
|  | 3,4-(HO)(HOOC)C <sub>6</sub> H <sub>3</sub> ,              | m. 205°. <sup>34</sup>                              |
|  | 2,4-Me <sub>2</sub> C <sub>6</sub> H <sub>3</sub> ,        | m. 155°. <sup>172</sup>                             |
|  | <i>o</i> -PhC <sub>6</sub> H <sub>4</sub> ,                | m. 174°. <sup>172</sup>                             |
|  | C <sub>10</sub> H <sub>7</sub> , α                         | m. 189°. <sup>172</sup> ; β m. 172°. <sup>172</sup> |

## R=Nitrophenyl

| R   | R'   |   |
|---|--|---|
| <i>o</i> -O <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> , | O <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> , <i>o</i> , | m. 160°. <sup>341</sup> ; 207°. <sup>1243</sup> <i>m</i> , m. 116°. <sup>341</sup><br>151°. <sup>1095</sup> <i>p</i> , m. 153°. <sup>341</sup>  |
|   | MeC <sub>6</sub> H <sub>4</sub> , <i>o</i> ,               | m. 192°. <sup>341</sup> <i>m</i> , m. 150°. <sup>341</sup> <i>p</i> , m.<br>207°. <sup>341</sup>  |
|   | 5,2-(O <sub>2</sub> N)MeC <sub>6</sub> H <sub>3</sub> ,    | m. 160°. <sup>341</sup>   |
|   | C <sub>10</sub> H <sub>7</sub> , α                         | m. 145°. <sup>341</sup> ; β m. 176°. <sup>341</sup>   |
| <i>m</i> -O <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> , | O <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> , <i>m</i> , | m. 161°. <sup>1243</sup> ; 167°. <sup>1095</sup> ; 168°. <sup>341</sup> <i>p</i> ,<br>m. 172°. <sup>341</sup> ; 184°. <sup>1095</sup>   |
|   | MeC <sub>6</sub> H <sub>4</sub> , <i>o</i> ,               | m. 166°. <sup>1095</sup> ; 154°. <sup>341</sup> <i>m</i> , m. 158°. <sup>341</sup><br>165°. <sup>1095</sup> <i>p</i> , m. 173°. <sup>341</sup> ,<br><sup>1095</sup> , <sup>1243</sup> |
|   | 3,4-BrMeC <sub>6</sub> H <sub>3</sub> ,                    | m. 143°. <sup>1095</sup>  |
|   | (O <sub>2</sub> N)MeC <sub>6</sub> H <sub>3</sub> , 4,2-,  | m. 188°. <sup>1243</sup> ; 158°. <sup>1368</sup> 5,2-,<br>m. 136°. <sup>341</sup> 2,4-, m. 188°,<br><sup>341</sup> 3,4-, m. 115°. <sup>341</sup>                                      |
|   | C <sub>10</sub> H <sub>7</sub> , α                         | m. 162°. <sup>1095</sup> ; 156°. <sup>341</sup> β m. 164°. <sup>341</sup><br>168°. <sup>1095</sup>  |
| <i>p</i> -O <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> , | HOOCCHMe,  | m. 185°. <sup>1000</sup>  |
|   | HOOCCH <sub>2</sub> CH <sub>2</sub> ,                      | m. 153°. <sup>386</sup> , <sup>1000</sup>   |
|   | HOOCCH <sub>2</sub> CHMe,                                  | m. 185°. <sup>386</sup>   |
|   | <i>p</i> -O <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> ,  | m. 175°. <sup>341</sup>   |

| R | R'  |
|---|---|
|   | MeC <sub>6</sub> H <sub>4</sub> , <i>o</i> , m. 132°; <sup>978</sup> <i>m</i> , m. 165°; <sup>341</sup> <i>p</i> , m. 165°. <sup>341</sup>                                  |
|   | (O <sub>2</sub> N)MeC <sub>6</sub> H <sub>3</sub> , 4,2-, m. 181°; <sup>341</sup> 5,2-, m. 175°; <sup>341</sup> 2,4-, m. 176°; <sup>341</sup> 3,4-, m. 176°. <sup>341</sup> |
|   | C <sub>10</sub> H <sub>7</sub> , $\alpha$ m. 187°; <sup>341</sup> $\beta$ m. 157°. <sup>341</sup>   |
|   | 3,4-(HO)(MeOOC)C <sub>6</sub> H <sub>3</sub> , m. 174°. <sup>34</sup>   |
|   | 3,5-(O <sub>2</sub> N) <sub>2</sub> C <sub>6</sub> H <sub>3</sub> , 3,5-(O <sub>2</sub> N) <sub>2</sub> C <sub>6</sub> H <sub>3</sub> , m. 160°. <sup>343</sup>             |

## R=Carboxyphenyl

| R   | R'  |
|---|---|
| <i>m</i> HOCC <sub>6</sub> H <sub>4</sub> ,             | <i>m</i> -HOCC <sub>6</sub> H <sub>4</sub> , diEt ester, dec. without melting. <sup>343</sup>   |
| <i>p</i> HOCC <sub>6</sub> H <sub>4</sub> ,             | <i>p</i> -HOCC <sub>6</sub> H <sub>4</sub> , diEt ester, m. 165°. <sup>343</sup>  |
|   | 3,4-(HOOC) <sub>2</sub> C <sub>6</sub> H <sub>3</sub> , m. 230°. <sup>384c</sup>  |
| 3,4-(HOOC) <sub>2</sub> C <sub>6</sub> H <sub>3</sub> , | 2-Pyridyl, m. 191°. <sup>384c</sup>   |
|   | 2-Thiazolyl, m. 185°. <sup>384c</sup>   |
| 3,4-(HO)(HOOC)C <sub>6</sub> H <sub>3</sub> ,           | MeC <sub>6</sub> H <sub>4</sub> , <i>o</i> , m. 181°; <sup>34</sup> <i>m</i> , m. 182°. <sup>34</sup>   |
|   | $\alpha$ -C <sub>10</sub> H <sub>7</sub> , m. 195°. <sup>1185</sup>   |
|   | 3,4-(HO)(HOOC)C <sub>6</sub> H <sub>3</sub> , m. 198° dec.; <sup>34</sup> 217°; <sup>34</sup> diMe ester, m. 179°; <sup>34</sup> Me ester, Et ester, m. 168°. <sup>34</sup> |
| 3,4-(HO)(MeOOC)C <sub>6</sub> H <sub>3</sub> ,          | Et <sub>2</sub> NCH <sub>2</sub> CH <sub>2</sub> , m. 181° dec. <sup>34</sup>   |
|   | 4,5,2-(HO)( <i>i</i> -Pr)MeC <sub>6</sub> H <sub>2</sub> , m. 187°. <sup>34</sup>   |
|   | 4-Amino-2-methyl-6-quinolyl, HCl, decomposes at 236°. <sup>34</sup>   |
|   | 2-Pyridyl, m. 212°. <sup>34</sup>   |
|   | 2,3-Dimethyl-1-phenyl-5-pyrazolin-4-yl, decomposes at 200°. <sup>34</sup>   |
|   | 2-Thiazolyl, m. 181°. <sup>34</sup>   |

## R=Phenyl with Various Substituents

| R   | R'   |
|---|--|
| <i>m</i> -NCC <sub>6</sub> H <sub>4</sub> ,   | <i>m</i> -NCC <sub>6</sub> H <sub>4</sub> , m. 144°; <sup>343</sup> 160°. <sup>589</sup> |
| <i>p</i> -NCC <sub>6</sub> H <sub>4</sub> ,   | <i>p</i> -NCC <sub>6</sub> H <sub>4</sub> , m. 171°; <sup>343</sup> 184°. <sup>589</sup> |
| <i>p</i> -NCSC <sub>6</sub> H <sub>4</sub> ,  | $\alpha$ -C <sub>10</sub> H <sub>7</sub> , m. 166°. <sup>169</sup>                       |
| <i>m</i> -MeCOC <sub>6</sub> H <sub>4</sub> , | <i>m</i> -MeCOC <sub>6</sub> H <sub>4</sub> , m. 135°. <sup>589</sup>                    |
| <i>p</i> -MeCOC <sub>6</sub> H <sub>4</sub> , | <i>p</i> -MeCOC <sub>6</sub> H <sub>4</sub> , m. 198°. <sup>343</sup>                    |

| R   | R'  |  |
|---|---|--|
| <i>m</i> -H <sub>2</sub> NSO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> , | <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> ,           | m. 161°. <sup>1185</sup>                                       |
|   | α-C <sub>10</sub> H <sub>7</sub> ,                    | m. 170°. <sup>1185</sup>                                       |
| <i>p</i> -H <sub>2</sub> NSO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> , | MeC <sub>6</sub> H <sub>4</sub> , <i>o</i> ,          | m. 195°; <sup>1327</sup> <i>m</i> , m. 210.5°; <sup>1327</sup> |
|   |   | <i>p</i> , m. 187.5°. <sup>1327</sup>                          |
|   | Me <sub>2</sub> C <sub>6</sub> H <sub>3</sub> , 2,4-, | m. 211°; <sup>1327</sup> 2,5-, m. 211°. <sup>1327</sup>        |
| <i>p</i> -Sulfamoyl C <sub>6</sub> H <sub>4</sub> ,                       | <i>p</i> -Sulfamoyl C <sub>6</sub> H <sub>4</sub> ,   | m. 248° dec. <sup>172</sup>                                    |
|   | <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> ,           | m. 234°. <sup>172</sup>  |
| <i>p</i> -(HO) <sub>2</sub> OAsC <sub>6</sub> H <sub>4</sub> ,            | <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> ,           | decomposes at 262°. <sup>172</sup>                             |

R=Tolyl

| R   | R'  |  |
|---|---|--|
| <i>o</i> -MeC <sub>6</sub> H <sub>4</sub> , | EtOCH <sub>2</sub> ,  | m. 128.5°. <sup>551</sup>  |
|   | MeC <sub>6</sub> H <sub>4</sub> , <i>o</i> ,                        | m. 158°; <sup>978, 1389</sup> 161°; <sup>592</sup> <i>m</i> , m. |
|   |   | 140°; <sup>978</sup> <i>p</i> , m. 132°. <sup>978</sup>          |
|   | 2,4-BrMeC <sub>6</sub> H <sub>3</sub>                               | m. 132°. <sup>978</sup>  |
|   | 4,2-(HO)MeC <sub>6</sub> H <sub>3</sub> ,                           | m. 182.5°. <sup>978</sup>  |
|   | (O <sub>2</sub> N)MeC <sub>6</sub> H <sub>3</sub> , 4,2-,           | m. 140°; <sup>341</sup> 5,2-, m.                                 |
|   |   | 149°; <sup>341</sup> 2,4-, m. 164°;                              |
|   |   | <sup>341</sup> 3,4-, m. 147°. <sup>341</sup>                     |
|   | 2,4-Me <sub>2</sub> C <sub>6</sub> H <sub>3</sub> ,                 | m. 143.5°. <sup>978</sup>  |
|   | 5,2-( <i>i</i> -Pr)MeC <sub>6</sub> H <sub>3</sub> ,                | m. 112°. <sup>810</sup>  |
|   | <i>p</i> -PhC <sub>6</sub> H <sub>4</sub> ,                         | m. 201°. <sup>141</sup>  |
|   | β-C <sub>10</sub> H <sub>7</sub> ,                                  | m. 194°. <sup>804</sup>  |
| <i>m</i> -MeC <sub>6</sub> H <sub>4</sub> , | Morpholinoethyl,  | m. 146°. <sup>173</sup>  |
|   | Morpholinopropyl,   | m. 98°. <sup>173</sup>   |
|   | <i>m</i> -MeC <sub>6</sub> H <sub>4</sub> ,                         | m. 112°. <sup>947</sup>  |
|   | 2,5-(MeO)MeC <sub>6</sub> H <sub>3</sub> ,                          | m. 129°. <sup>172</sup>  |
|   | (O <sub>2</sub> N)MeC <sub>6</sub> H <sub>3</sub> , 4,2-,           | m. 152°; <sup>341</sup> 5,2-, m.                                 |
|   |   | 156°; <sup>341</sup> 2,4-, m. 156°;                              |
|   |   | <sup>341</sup> 3,4-, m. 152°. <sup>341</sup>                     |
|   | <i>p</i> -EtC <sub>6</sub> H <sub>4</sub> ,                         | m. 98°. <sup>172</sup>   |
|   | 5,2-( <i>i</i> -Pr)MeC <sub>6</sub> H <sub>3</sub> ,                | m. 101°. <sup>810</sup>  |
|   | 2-Pyridyl,  | m. 170°; <sup>1082</sup> 174°. <sup>172</sup>                    |
| <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> , | MeOCH <sub>2</sub> ,  | m. 129°. <sup>874</sup>  |
|   | EtOCH <sub>2</sub> ,  | m. 120°. <sup>874</sup>  |
|   | <i>i</i> -AmOCH <sub>2</sub> ,                                      | m. 119°. <sup>874</sup>  |
|   | MeSO <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> , | m. 150°. <sup>732</sup>  |
|   | MeSO <sub>2</sub> (CH <sub>2</sub> ) <sub>4</sub> ,                 | m. 108°. <sup>724</sup>  |

| R   | R'  |
|---|---|
|   | Morpholinoethyl, m. 149°. <sup>173</sup>  |
|   | Morpholinopropyl, m. 106°. <sup>173</sup>   |
|   | <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> , m. 174°. <sup>947</sup> 178°. <sup>1389</sup>                   |
|   | ClMeC <sub>6</sub> H <sub>3</sub> , 3,2-, m. 180°; <sup>153</sup> 5,3-, m. 156°; <sup>153</sup>             |
|   | 3,4-, m. 160°. <sup>153</sup>   |
|   | (O <sub>2</sub> N)MeC <sub>6</sub> H <sub>3</sub> , 4,2-, m. 150°; <sup>341</sup> 5,2-, m.                  |
|   | 161°; <sup>341</sup> 6,2-, m. 160°;   |
|   | <sup>341</sup> 4,3-, m. 110°; <sup>341</sup>  |
|   | 2,4-, m. 162°; <sup>341</sup> 169°;   |
|   | <sup>1243</sup> 3,4-, m. 176°. <sup>341</sup>   |
|   | Me <sub>2</sub> C <sub>6</sub> H <sub>3</sub> , 2,3-, m. 156°; <sup>172</sup> 2,5-, m. 128°; <sup>172</sup> |
|   | 3,4-, m. 134°. <sup>172</sup>   |
|   | EtC <sub>6</sub> H <sub>4</sub> , <i>o</i> , m. 182°; <sup>172</sup> <i>p</i> , m.                          |
|   | 155°. <sup>172</sup>  |
|   | <i>p</i> -PrC <sub>6</sub> H <sub>4</sub> , m. 131°. <sup>172</sup>   |
|   | 5,2-( <i>i</i> -Pr)MeC <sub>6</sub> H <sub>3</sub> , m. 116°. <sup>810</sup>                                |
|   | <i>p</i> -BuC <sub>6</sub> H <sub>4</sub> , m. 137°. <sup>172</sup>   |
|   | <i>p</i> -HepC <sub>6</sub> H <sub>4</sub> , m. 129°. <sup>172</sup>  |
|   | <i>p</i> -PhC <sub>6</sub> H <sub>4</sub> , m. 192°. <sup>141</sup>   |
|   | C <sub>10</sub> H <sub>7</sub> , $\alpha$ m. 168°; <sup>864</sup> 204°; <sup>172</sup>                      |
|   | $\beta$ m. 164°; <sup>864</sup> 171°. <sup>172</sup>  |
|   | 2-Pyridyl, m. 182°; <sup>1082</sup> 192°. <sup>172</sup>  |
|   | Amidino, m. 200°; <sup>780b</sup> picrate, m.   |
|   | 268°. <sup>780b</sup>   |
|   | 2-Thiazolyl, m. 201°. <sup>172</sup>  |
| 2,3-ClMeC <sub>6</sub> H <sub>3</sub> ,                 | $\beta$ -C <sub>10</sub> H <sub>7</sub> , m. 172°. <sup>153</sup>   |
| 2,4-ClMeC <sub>6</sub> H <sub>3</sub> ,                 | $\beta$ -C <sub>10</sub> H <sub>7</sub> , m. 149°. <sup>153</sup>   |
| 2,5-ClMeC <sub>6</sub> H <sub>3</sub> ,                 | $\beta$ -C <sub>10</sub> H <sub>7</sub> , m. 154°. <sup>153</sup>   |
| 4,3-ClMeC <sub>6</sub> H <sub>3</sub> ,                 | $\beta$ -C <sub>10</sub> H <sub>7</sub> , m. 158°. <sup>153</sup>   |
| 5,2-ClMeC <sub>6</sub> H <sub>3</sub> ,                 | $\beta$ -C <sub>10</sub> H <sub>7</sub> , m. 163°. <sup>153</sup>   |
| 6,2-ClMeC <sub>6</sub> H <sub>3</sub> ,                 | $\beta$ -C <sub>10</sub> H <sub>7</sub> , m. 150°. <sup>153</sup>   |
| 5,2-BrMeC <sub>6</sub> H <sub>3</sub> ,                 | 5,2-BrMeC <sub>6</sub> H <sub>3</sub> , m. 159°. <sup>342</sup>   |
| 2,5-(MeO)MeC <sub>6</sub> H <sub>3</sub> ,              | 2,5-(MeO)MeC <sub>6</sub> H <sub>3</sub> , m. 144°; <sup>1033</sup> 146°. <sup>172</sup>                    |
| 4,3-(EtO)MeC <sub>6</sub> H <sub>3</sub> ,              | 4,3-(EtO)MeC <sub>6</sub> H <sub>3</sub> , m. 162°. <sup>583</sup>  |
| 3,2-(O <sub>2</sub> N)MeC <sub>6</sub> H <sub>3</sub> , | $\alpha$ -C <sub>10</sub> H <sub>7</sub> , m. 171°. <sup>341</sup>  |
| 4,2-(O <sub>2</sub> N)MeC <sub>6</sub> H <sub>3</sub> , | C <sub>10</sub> H <sub>7</sub> , $\alpha$ m. 191°; <sup>341</sup> $\beta$ m. 154°.                          |
|   | <sup>341</sup>  |
| 5,2-(O <sub>2</sub> N)MeC <sub>6</sub> H <sub>3</sub> , | 5,2-(O <sub>2</sub> N)MeC <sub>6</sub> H <sub>3</sub> , m. 131°; <sup>342</sup> 162°. <sup>341</sup>        |
|   | C <sub>10</sub> H <sub>7</sub> , $\alpha$ m. 166°; <sup>341</sup> $\beta$ m. 165°.                          |
|   | <sup>341</sup>  |

| R   | R' |   |
|---|----|---|
| 2,4-(O <sub>2</sub> N)MeC <sub>6</sub> H <sub>3</sub> , C <sub>10</sub> H <sub>7</sub> ,                        |    | $\alpha$ m. 165°; <sup>341</sup> $\beta$ m. 212°. |
|   |    | <sup>341</sup>                                    |
| 3,4-(O <sub>2</sub> N)MeC <sub>6</sub> H <sub>3</sub> , C <sub>10</sub> H <sub>7</sub> ,                        |    | $\alpha$ m. 168°; <sup>341</sup> $\beta$ m. 159°. |
|   |    | <sup>341</sup>                                    |
| 2,5-(O <sub>2</sub> N)MeC <sub>6</sub> H <sub>3</sub> , 2,5-(O <sub>2</sub> N)MeC <sub>6</sub> H <sub>3</sub> , |    | m. 172°. <sup>341</sup>                           |
| $\alpha$ -C <sub>10</sub> H <sub>7</sub> ,  |    | m. 142°. <sup>341</sup>                           |

## R=Higher Aryl

| R   | R'  |  |
|---|---|--|
| 2,3-Me <sub>2</sub> C <sub>6</sub> H <sub>3</sub> ,   | 2,3-Me <sub>2</sub> C <sub>6</sub> H <sub>3</sub> ,   | m. 224°. <sup>172</sup>                        |
| 2,4-Me <sub>2</sub> C <sub>6</sub> H <sub>3</sub> ,   | Morpholinopropyl,   | m. 115°. <sup>178</sup>                        |
|   | 2,4-Me <sub>2</sub> C <sub>6</sub> H <sub>3</sub> ,   | m. 150°. <sup>342</sup>                        |
|   | <i>p</i> -PrC <sub>6</sub> H <sub>4</sub> ,   | m. 129°. <sup>172</sup>                        |
|   | C <sub>10</sub> H <sub>7</sub> , $\alpha$ m. 189°; <sup>172</sup> $\beta$ m. 168°. <sup>172</sup> |  |
|   | 2-Pyridyl,  | m. 193°. <sup>172</sup>                        |
|   | 2-Thiazolyl,  | m. 204°. <sup>172</sup>                        |
| Me <sub>2</sub> C <sub>6</sub> H <sub>3</sub> ,   | Me <sub>2</sub> C <sub>6</sub> H <sub>3</sub> , (2,5-) <sub>2</sub> ,                             | m. 148°. <sup>947</sup> 148.5°; <sup>342</sup> |
|   |   | (2,6-) <sub>2</sub> , m. 208°; <sup>343</sup>  |
|   |   | (3,4-) <sub>2</sub> , m. 179°. <sup>342</sup>  |
| 2,3,5-ClMe <sub>2</sub> C <sub>6</sub> H <sub>2</sub> , $\beta$ -C <sub>10</sub> H <sub>7</sub> , |   | m. 154°. <sup>153</sup>                        |
| <i>p</i> -EtC <sub>6</sub> H <sub>4</sub> ,   | <i>p</i> -PrC <sub>6</sub> H <sub>4</sub> ,   | m. 139°. <sup>172</sup>                        |
|   | <i>p</i> -BuC <sub>6</sub> H <sub>4</sub> ,   | m. 138°. <sup>172</sup>                        |
|   | C <sub>10</sub> H <sub>7</sub> , $\alpha$ m. 173°; <sup>172</sup> $\beta$ m. 183°. <sup>172</sup> |  |
| 2,4,5-Me <sub>3</sub> C <sub>6</sub> H <sub>2</sub> ,   | 2,4,5-Me <sub>3</sub> C <sub>6</sub> H <sub>2</sub> ,   | m. 146.5°. <sup>342</sup>                      |
| 3,2,4,6-ClMe <sub>3</sub> C <sub>6</sub> H,   | $\beta$ -C <sub>10</sub> H <sub>7</sub> ,   | m. 181°. <sup>153</sup>                        |
| 3,2,4,6-(O <sub>2</sub> N)Me <sub>3</sub> C <sub>6</sub> H,                                       | 3,2,4,6-(O <sub>2</sub> N)Me <sub>3</sub> C <sub>6</sub> H,                                       | m. 244°. <sup>342</sup>                        |
| <i>p</i> -PrC <sub>6</sub> H <sub>4</sub> ,   | <i>p</i> -PrC <sub>6</sub> H <sub>4</sub> ,   | m. 149°. <sup>133</sup> 154°. <sup>172</sup>   |
|   | <i>p</i> -BuC <sub>6</sub> H <sub>4</sub> ,   | m. 164°. <sup>172</sup>                        |
|   | <i>p</i> -HepC <sub>6</sub> H <sub>4</sub> ,  | m. 138°. <sup>172</sup>                        |
|   | <i>o</i> -PhC <sub>6</sub> H <sub>4</sub> ,   | m. 143°. <sup>172</sup>                        |
|   | $\beta$ -C <sub>10</sub> H <sub>7</sub> ,   | m. 179°. <sup>172</sup>                        |
|   | 2-Thiazolyl,  | m. 204°. <sup>172</sup>                        |
| 2,5-(Me) ( <i>i</i> -Pr)C <sub>6</sub> H <sub>3</sub> ,   | 2,5-(Me) ( <i>i</i> -Pr)C <sub>6</sub> H <sub>3</sub> ,   | m. 130°. <sup>810</sup>                        |
| <i>p</i> -BuC <sub>6</sub> H <sub>4</sub> ,   | <i>p</i> -BuC <sub>6</sub> H <sub>4</sub> ,   | m. 150°. <sup>583</sup> 159°. <sup>172</sup>   |
|   | <i>p</i> -HepC <sub>6</sub> H <sub>4</sub> ,  | m. 140°. <sup>172</sup>                        |
|   | $\beta$ -C <sub>10</sub> H <sub>7</sub> ,   | m. 179°. <sup>172</sup>                        |
|   | 2-Pyridyl,  | m. 156°. <sup>172</sup>                        |
| <i>o</i> -Me <sub>3</sub> CC <sub>6</sub> H <sub>4</sub> ,  | <i>o</i> -Me <sub>3</sub> CC <sub>6</sub> H <sub>4</sub> ,  | m. 182°. <sup>172</sup>                        |
|   | $\beta$ -C <sub>10</sub> H <sub>7</sub> ,   | m. 176°. <sup>172</sup>                        |
| <i>p</i> -Me <sub>3</sub> CC <sub>6</sub> H <sub>4</sub> ,  | <i>p</i> -Me <sub>3</sub> CC <sub>6</sub> H <sub>4</sub> ,  | m. 193°. <sup>583</sup>                        |

| R   | R'  |  |
|---|---|--|
| $p\text{-HepC}_6\text{H}_4$ ,                               | $p\text{-HepC}_6\text{H}_4$ ,                               | m. $152^\circ$ . <sup>172</sup>  |
|   | $p\text{-PhC}_6\text{H}_4$ ,                                | m. $219^\circ$ . <sup>172</sup>  |
|   | $\text{C}_{10}\text{H}_7$ , $\alpha$                        | m. $149^\circ$ ; <sup>172</sup> $\beta$ m. $176^\circ$ . <sup>172</sup>  |
| $(p\text{-ClC}_6\text{H}_4)_2\text{CH}$ ,                   | $(p\text{-ClC}_6\text{H}_4)_2\text{CH}$ ,                   | m. $211^\circ$ . <sup>1418</sup>   |
|   | $(p\text{-EtOC}_6\text{H}_4)_2\text{CH}$ ,                  | m. $170^\circ$ . <sup>1418</sup>   |
| $(p\text{-EtOC}_6\text{H}_4)_2\text{CH}$ ,                  | $(p\text{-EtOC}_6\text{H}_4)_2\text{CH}$ ,                  | m. $180^\circ$ . <sup>1418</sup>   |
| $o\text{-PhC}_6\text{H}_4$ ,                                | $o\text{-PhC}_6\text{H}_4$ ,                                | m. $155^\circ$ ; <sup>1033</sup> $166^\circ$ . <sup>172</sup>  |
|   | $\beta\text{-C}_{10}\text{H}_7$ ,                           | m. $192^\circ$ . <sup>172</sup>  |
|   | $\alpha\text{-[4-ClC}_{10}\text{H}_6]$ ,                    | m. $198^\circ$ . <sup>172</sup>  |
|   | 2-Pyridyl,  | m. $176^\circ$ . <sup>172</sup>  |
| $p\text{-PhC}_6\text{H}_4$ ,                                | Morpholinoethyl,  | m. $164^\circ$ . <sup>173</sup>  |
|   | Morpholinopropyl,   | m. $106^\circ$ . <sup>173</sup>  |
|   | $\alpha\text{-Ph-}\gamma\text{-(2-piperidyl)butyl}$ ,       | m. $81^\circ$ . <sup>111</sup>   |
|   | $p\text{-PhC}_6\text{H}_4$ ,                                | m. $230^\circ$ , <sup>1033</sup> $235^\circ$ . <sup>150</sup>  |
|   | Bornyl,   | m. $167^\circ$ . <sup>150</sup>  |
|   | Camphyl,  | m. $138^\circ$ . <sup>150</sup>  |
|   | $\beta\text{-C}_{10}\text{H}_7$ ,                           | m. $242^\circ$ . <sup>172</sup>  |
| $o\text{-(}p\text{-ClC}_6\text{H}_4\text{)C}_6\text{H}_4$ , | $o\text{-(}p\text{-ClC}_6\text{H}_4\text{)C}_6\text{H}_4$ , | m. $193^\circ$ . <sup>1033</sup>   |
| $p\text{-(}p\text{-ClC}_6\text{H}_4\text{)C}_6\text{H}_4$ , | $p\text{-(}p\text{-ClC}_6\text{H}_4\text{)C}_6\text{H}_4$ , | m. $220^\circ$ . <sup>1033</sup>   |
| $4,2,5\text{-BrMePhC}_6\text{H}_2$ ,                        | $4,2,5\text{-BrMePhC}_6\text{H}_2$ ,                        | m. $176^\circ$ . <sup>1033</sup>   |
| $\alpha\text{-C}_{10}\text{H}_7$ ,                          | Morpholinoethyl,  | m. $142^\circ$ . <sup>173</sup>  |
|   | $\text{MeSO}_2\text{CH}_2\text{CH}_2\text{CH}_2$ ,          | m. $183^\circ$ . <sup>732</sup>  |
|   | $\text{MeSO}_2(\text{CH}_2)_4$ ,                            | m. $163^\circ$ . <sup>724</sup>  |
|   | $\text{ClC}_6\text{H}_4\text{CH}_2$ , $o$ ,                 | m. $157^\circ$ ; <sup>1167</sup> $m$ , m. $124^\circ$ ; <sup>1167</sup> $p$ , m. $143^\circ$ . <sup>1167</sup> |
|   | $\text{BrC}_6\text{H}_4\text{CH}_2$ , $o$ ,                 | m. $163^\circ$ ; <sup>1167</sup> $m$ , m. $129^\circ$ ; <sup>1167</sup> $p$ , m. $157^\circ$ . <sup>1167</sup> |
|   | $\text{IC}_6\text{H}_4\text{CH}_2$ , $m$ ,                  | m. $156^\circ$ ; <sup>1167</sup> $p$ , m. $166^\circ$ . <sup>1167</sup>  |
|   | $\text{C}_{10}\text{H}_7$ , $\alpha$                        | m. $203^\circ$ ; <sup>947</sup> $\beta$ m. $201^\circ$ . <sup>172</sup>  |
|   | $\alpha\text{-(4-ClC}_{10}\text{H}_6)$ ,                    | m. $212^\circ$ . <sup>172</sup>  |
|   | $\alpha\text{-(5-HOC}_{10}\text{H}_6)$ ,                    | m. $219^\circ$ . <sup>172</sup>  |
|   | 2-Pyridyl,  | m. $198^\circ$ , <sup>1185</sup> $236^\circ$ . <sup>172</sup>  |
|   | 6-Methyl-2-pyridyl,   | m. $212^\circ$ . <sup>172</sup>  |
|   | 2-Thiazolyl,  | m. $235^\circ$ . <sup>172</sup>  |
|   | 2-(4-Methylthiazolyl),                                      | m. $190^\circ$ . <sup>1185</sup>   |
|   | Morpholinyl,  | m. $163^\circ$ . <sup>996</sup>  |
| $\beta\text{-C}_{10}\text{H}_7$ ,                           | $\text{ClC}_6\text{H}_4\text{CH}_2$ , $o$ ,                 | m. $160^\circ$ ; <sup>1167</sup> $m$ , m. $153^\circ$ ; <sup>1167</sup> $p$ , m. $169^\circ$ . <sup>1167</sup> |

| R  | R'  |
|--|---|
|  | BrC <sub>6</sub> H <sub>4</sub> CH <sub>2</sub> , <i>o</i> , m. 161°; <sup>1167</sup> <i>m</i> , m. 163°; <sup>1167</sup> <i>p</i> , m. 173°. <sup>1167</sup> |
|  | IC <sub>6</sub> H <sub>4</sub> CH <sub>2</sub> , <i>m</i> , m. 172°; <sup>1167</sup> <i>p</i> , m. 180°. <sup>1167</sup>                                      |
|  | β-C <sub>10</sub> H <sub>7</sub> , m. 193°; <sup>245</sup> 202°. <sup>947</sup>   |
|  | Camphyl, m. 127°. <sup>150</sup>  |
|  | 2-Pyridyl, m. 242°. <sup>172</sup>  |
|  | 6-Methyl-2-pyridyl, m. 252°. <sup>172</sup>   |
|  | 2-Thiazolyl, m. 232°. <sup>172</sup>  |
| α-(4-ClC <sub>10</sub> H <sub>6</sub> ), | β-C <sub>10</sub> H <sub>7</sub> , m. 189°. <sup>172</sup>  |
|  | α-[4-ClC <sub>10</sub> H <sub>6</sub> ], m. 202°. <sup>172</sup>  |

## R and R'=Heterocyclic

| R                      | R'  |
|------------------------|---|
| 1-Anthranyl,           | 1-Anthranyl, m. 234°. <sup>67</sup>               |
| 2-Anthranyl,           | 2-Anthranyl, m. 262°. <sup>67</sup>               |
| 2-Quinolyl,            | 2-Quinolyl, m. 214°. <sup>37</sup>                |
| 6-Quinolyl,            | 6-Quinolyl, m. 218°. <sup>644</sup>               |
| 4-Amino-6-quinolyl,    | 4-Amino-6-quinolyl, m. 235°. <sup>924</sup>       |
| 1,2-Dihydroindan-5-yl, | 1,2-Dihydroindan-5-yl, m. 128°. <sup>172</sup>    |
| 4-Amino-6-cinnolyl,    | 4-Amino-6-cinnolyl, 2HCl, m. 270°. <sup>924</sup> |
| 3-Pyridyl,             | 3-Pyridyl, m. 176°. <sup>381</sup>                |
| Xanthyl,               | Xanthyl, m. 200° dec. <sup>415</sup>              |
| 2-Thiazolyl,           | 2-Thiazolyl, m. 260°. <sup>172</sup>              |

## 1,3,3-TRISUBSTITUTED THIOUREAS, RNHCSNR'R''

| R   | R'                                   | R''   |
|-----|--------------------------------------|---|
| Me, | Me,                                  | Me, m. 87°; <sup>801, 809, 1195</sup> 88°. <sup>305c</sup>                      |
|     |                                      | CN, m. 195° dec. <sup>541</sup>   |
|     |                                      | PhCH <sub>2</sub> , m. 86°. <sup>577</sup>                                      |
|     |                                      | 3,4-(HO)(HOOC)C <sub>6</sub> H <sub>3</sub> , decomposes at 126°. <sup>34</sup> |
|     |                                      | <i>p</i> -HOCC <sub>6</sub> H <sub>4</sub> , m. 163°. <sup>34</sup>             |
|     | Et,                                  | Et, m. 28°. <sup>762</sup>  |
|     | Pr,                                  | CN, m. 90.5°. <sup>541</sup>  |
|     | CH <sub>2</sub> :CHCH <sub>2</sub> , | m. 77.5°. <sup>541</sup>  |
|     | PhCH <sub>2</sub> ,                  | m. 173°. <sup>541</sup>   |



| R                                    | R'                                   | R''   |               |
|--------------------------------------|--------------------------------------|---|---------------|
| <i>i</i> -AmOCH <sub>2</sub> ,       | Me,                                  | Ph,   | m. 87°.674    |
| Et,                                  | Me,                                  | Me,   | m. 37.5°.96   |
|                                      |                                      | Et,   | m. 37°.805    |
|                                      | Et,                                  |   | m. 146°.1025  |
|                                      |                                      | CN,   | m. 98.2°.541  |
|                                      | Pr,                                  | Pr,   | m. 34.5°.96   |
|                                      |                                      | CN,   | m. 74.7°.541  |
|                                      | CH <sub>2</sub> :CHCH <sub>2</sub> , |   | m. 81.2°.541  |
|                                      | PhCH <sub>2</sub> ,                  |   | m. 143.5°.541 |
| HOCH <sub>2</sub> CH <sub>2</sub> ,  | Ph,                                  | Ph,   | m. 108°.263   |
| Pr,                                  | Me,                                  | CN,   | m. 115°.541   |
|                                      | Et,                                  |   | m. 56°.541    |
|                                      | Pr,                                  | Pr,   | m. 33°.281    |
|                                      |                                      | CN,   | m. 56°.281    |
|                                      | CH <sub>2</sub> :CHCH <sub>2</sub> , |   | m. 50.3°.541  |
|                                      | PhCH <sub>2</sub> ,                  |   | m. 113°.541   |
| <i>s</i> -Bu                         | Me,                                  | Me,   | m. 54°.1308   |
| Oct,                                 | Me,                                  | Me  | m. 75°.121    |
| CH <sub>2</sub> :CHCH <sub>2</sub> , | Et,                                  | Et,   | m. 55°.452    |
|                                      |                                      | CN,   | m. 63.2°.541  |
|                                      | Pr,                                  |   | m. 57.3°.541  |
|                                      | CH <sub>2</sub> :CHCH <sub>2</sub> , |   | m. 52.4°.541  |
|                                      | PhCH <sub>2</sub> ,                  |   | m. 116°.541   |
| PhCMe <sub>2</sub> ,                 | Me,                                  | Me,   | m. 75°.841    |
| PhCH <sub>2</sub> CH <sub>2</sub> ,  |                                      |   | m. 112°.184   |
| Ph,                                  |                                      | Butadienyl-2,3,                             | m. 63.5°.367  |
|                                      |                                      | <i>o</i> -MeC <sub>6</sub> H <sub>4</sub> , | m. 90°.435    |
|                                      |                                      | <i>m</i> -MeC <sub>6</sub> H <sub>4</sub> , | m. 67°.435    |
|                                      |                                      | <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> , | m. 89.4°.435  |
|                                      |                                      | 5-Hydroxymethyl-2-thenyl,                   | m. 101°.534   |
|                                      | Et,                                  | PhCH <sub>2</sub> ,                         | m. 95°.563    |
|                                      | HOCH <sub>2</sub> CH <sub>2</sub> ,  | Ph,   | m. 108°.263   |
|                                      | Pr,                                  | MeOOC,                                      | m. 83°.308a   |
|                                      | <i>i</i> -Bu,                        | CN,   | m. 139°.541   |
|                                      | CH <sub>2</sub> :CHCH <sub>2</sub> , | Ph,   | m. 91°.262    |
|                                      |                                      | <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> , | m. 91.5°.262  |
|                                      | Cyclohexyl,                          | Cyclohexyl,                                 | m. 89°.182    |

| R  | R'   | R''  |  |
|--|--|--|--|
|  | Ph,  | MeOOC,<br>2-Furfuryl,                        | m. 106°. <sup>308a</sup><br>m. 76° and<br>110°. <sup>519</sup>               |
|  | PhCH <sub>2</sub> ,<br><i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> ,<br><i>p</i> -MeC <sub>6</sub> H <sub>4</sub> , | PhCH <sub>2</sub> ,<br>2-Furfuryl,           | m. 145°. <sup>182</sup><br>m. 99°. <sup>519</sup><br>m. 119°. <sup>519</sup> |
| PhCHMe,<br>PhCH <sub>2</sub> CH <sub>2</sub> ,<br>PhCMe <sub>2</sub> ,<br><i>p</i> -BrC <sub>6</sub> H <sub>4</sub> ,                                | Me,  | Me,  | m. 109°. <sup>841</sup><br>m. 112°. <sup>134</sup><br>m. 75°. <sup>841</sup> |
| <i>p</i> -BrC <sub>6</sub> H <sub>4</sub> ,<br>3-4- (HO) (MeOOC) C <sub>6</sub> H <sub>3</sub> , Me,<br><i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> , | CH <sub>2</sub> :CHCH <sub>2</sub> ,<br>Ph,  | Ph,  | m. 123°. <sup>262</sup><br>m. 140°. <sup>34</sup><br>m. 103°. <sup>182</sup> |
|  | Et,  | Et,  | m. 105°. <sup>182</sup>  |
|  | Bu,  | Ph,  | m. 99°. <sup>182</sup>   |
|  | Cyclohexyl,  | Bu,  | m. 82°. <sup>182</sup>   |
|  | PhCH <sub>2</sub> ,  | Cyclohexyl,                                  | m. 89°. <sup>182</sup>   |
| <i>p</i> -EtOC <sub>6</sub> H <sub>4</sub> ,   | Me,  | PhCH <sub>2</sub> ,                          | m. 117°. <sup>182</sup>  |
| 3,4- (HOOC) <sub>2</sub> C <sub>6</sub> H <sub>3</sub> ,   |  | <i>p</i> -EtOC <sub>6</sub> H <sub>4</sub> , | m. 59°. <sup>583</sup>   |
| <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> ,  | CH <sub>2</sub> :CHCH <sub>2</sub> ,   | HOOCCH <sub>2</sub> ,                        | m. 247° dec. <sup>384c</sup>   |
| <i>p</i> -PhC <sub>6</sub> H <sub>4</sub> ,  | Me,  | Ph,  | m. 107°. <sup>262</sup>  |
|  | Et,  | Me,  | m. 225°. <sup>150</sup>  |
| <i>p</i> -PhC <sub>6</sub> H <sub>4</sub> ,  | Pr,  | Et,  | m. 114°. <sup>150</sup>  |
|  | <i>i</i> -Bu,  | Pr,  | m. 117°. <sup>150</sup>  |
|  | Am,  | <i>i</i> -Bu,                                | m. 160°. <sup>150</sup>  |
| $\alpha$ -C <sub>10</sub> H <sub>7</sub> ,   | Me,  | Am,  | m. 118°. <sup>150</sup>  |
|  |  | Me,  | m. 165°. <sup>996</sup><br>168°. <sup>1262</sup>                             |
|  | Et,  | Et,  | m. 108°. <sup>1262</sup>   |
|  | Pr,  | Pr,  | m. 161°. <sup>1262</sup>   |
|  | Bu,  | Bu,  | m. 123°. <sup>1262</sup>   |
|  | <i>i</i> -Am,  | <i>i</i> -Am,                                | m. 118°. <sup>1262</sup>   |
|  | PhCH <sub>2</sub> ,  | PhCH <sub>2</sub> ,                          | m. 101°. <sup>1262</sup>   |
| $\beta$ -C <sub>10</sub> H <sub>7</sub> ,  | Me,  | Me,  | m. 173°. <sup>150</sup>  |
|  |  | $\beta$ -C <sub>10</sub> H <sub>7</sub> ,    | m. 178°. <sup>593</sup>  |
|  | Et,  | Et,  | m. 90°. <sup>150</sup>   |
|  | Pr,  | Pr,  | m. 109°. <sup>150</sup>  |
|  | <i>i</i> -Bu,  | <i>i</i> -Bu,                                | m. 136°. <sup>150</sup>  |
|  | Am,  | Am,  | m. 126°. <sup>150</sup>  |
| EtOOC,   | Ph,  | Ph,  | m. 128°. <sup>553</sup>  |

## 1-R-4-R'-5-TETRAZOLYL:NCSNHPh 550, 552, 553

|               |   |
|---------------|---|
| R=Methyl,     | R'=Ethyl, m. 150                              |
|               | =i-Butyl, m. 112°.                            |
|               | =Heptyl, m. 77°.                              |
|               | =Cyclohexyl, m. 173°.                         |
|               | =Phenyl, m. 182°.                             |
| R=Ethyl,      | =Benzyl, m. 124°.                             |
|               | R'=Propyl, m. 78°.                            |
|               | =i-Propyl, m. 110°.                           |
|               | =Butyl, m. 73°.                               |
|               | =i-Butyl, m. 78°.                             |
|               | =Amyl, m. 78°.                                |
|               | =i-Amyl, m. 83°.                              |
|               | =3-Amyl, m. 121°.                             |
|               | =Cyclohexyl, m. 144°.                         |
|               | =Phenyl, m. 94°.                              |
| R=Cyclohexyl, | =Benzyl, m. 118°.                             |
|               | =PhCH <sub>2</sub> CH <sub>2</sub> , m. 82°.  |
|               | R'=Benzyl, m. 151°.                           |
|               | =PhCH <sub>2</sub> CH <sub>2</sub> , m. 121°. |

## 1,1,3,3-TETRASUBSTITUTED THIOUREAS

| R   | R'  | R'' | R''' |   |
|-----|-----|-----|------|---|
| Me, | Me, | Me, | Me,  | m. 78°; <sup>281</sup> , <sup>1099</sup> b. 245°. <sup>281</sup>  |
| Me, | Me, | Et, | Et,  | m. 37.5°. <sup>96</sup>   |
| Me, | Et, | Me, | Et,  | b <sub>12</sub> 125°. <sup>804</sup>  |
| Et, | Et, | Et, | Et,  | b <sub>12</sub> 130°; <sup>281</sup> b. 266°; <sup>281</sup> 267°; <sup>1099</sup><br>d. 0/4 0.9804; <sup>281</sup> d. 18/4<br>0.9662; <sup>281</sup> n <sub>D</sub> <sup>20</sup> 1.5225. <sup>281</sup> |
| Pr, | Pr, | Pr, | Pr,  | b <sub>12</sub> 165°; <sup>281</sup> b. 305°; <sup>281</sup> d. 0/4<br>0.9430; <sup>281</sup> d 19/4 0.9300; <sup>281</sup><br>n <sub>D</sub> <sup>22</sup> 1.5079. <sup>281</sup>                        |

## BIS-THIOUREAS

|   |   |
|---|---|
| CH <sub>2</sub> [NHCSNH <sub>2</sub> ] <sub>2</sub> ,   | m. 252°; <sup>298</sup><br>272°. <sup>1237b</sup> |
| CH <sub>2</sub> [NHCSNH <sub>2</sub> Et] <sub>2</sub> , | m. 132°. <sup>944</sup>                           |

|   |                                  |
|---|----------------------------------|
| $\text{CH}_2[\text{NHCSNHAc}]_2$ ,  | m. 167°. <sup>299</sup>          |
| $\text{CH}_2[\text{NMeCSNHMe}]_2$ ,   | m. 168°. <sup>1237b</sup>        |
| $\text{MeCH}(\text{NHCSNH}_2)_2$ ,  | m. 212° dec. <sup>308c</sup>     |
| $\text{H}_2\text{NCSNHCH}_2\text{CH}_2\text{NHCSN}(\text{Et})_2$ ,                            | m. 130°. <sup>1025</sup>         |
| $[\cdot\text{CH}_2\text{NHCSNHCH}_2\text{CH}:\text{CH}_2]_2$ ,                                | m. 102°. <sup>432</sup>          |
| $[\cdot\text{CH}_2\text{NHCSNHPh}]_2$ ,   | m. 190°. <sup>182</sup>          |
| $[\cdot\text{CH}_2\text{NHCSNHC}_6\text{H}_4\text{OMe-}p]_2$ ,                                | m. 190° dec. <sup>182</sup>      |
| $[\cdot\text{CH}_2\text{NHCSNHC}_6\text{H}_4\text{Ph-}p]_2$ ,                                 | m. 237°. <sup>150</sup>          |
| $[\cdot\text{CH}_2\text{NHCSNHC}_{10}\text{H}_7\text{-}\beta]_2$ ,                            | m. 223°. <sup>150</sup>          |
| $\text{CH}_2[\text{CH}_2\text{NHCSNH}_2]_2$ ,   | m. 179°. <sup>651</sup>          |
| $\text{H}_2\text{NCSNHCH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{NHCSN}(\text{Et})_2$ ,      | m. 174°. <sup>1025</sup>         |
| $[\cdot\text{CH}_2\text{CH}_2\text{NHCSN}(\text{CH}_2)_6]_2$ ,                                | m. 131°. <sup>652</sup>          |
| 2,3- $[\cdot\text{CMe}_2\text{NHCSNH}_2]_2$ ,   | m. 243°. <sup>543</sup>          |
| $[\cdot\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{NHCSNH}_2]_2$ ,           | m. 175°. <sup>1409</sup>         |
| 1,3- $[\text{NH}_2\text{CSNH}]_2\text{C}_6\text{H}_4$ ,                                       | m. 215°. <sup>97</sup>           |
| 1,4- $[\text{NH}_2\text{CSNH}]_2\text{C}_6\text{H}_4$ ,                                       | m. 220°. <sup>97</sup>           |
| 1,4- $\text{H}_2\text{NCSNHC}_6\text{H}_4\text{NHCSNH}(\text{i-Pr})$ ,                        | m. 201°. <sup>446</sup>          |
| 1,4- $\text{H}_2\text{NCSNHC}_6\text{H}_4\text{NHCSNHC}_6\text{H}_4\text{Cl-}p$ ,             | m. 239°. <sup>446</sup>          |
| 1,4- $[(\text{iPr})\text{NHCSNH}]_2\text{C}_6\text{H}_4$ ,                                    | m. 221°. <sup>446</sup>          |
| 1,4- $(\text{i-Pr})\text{NHCSNHC}_6\text{H}_4\text{NHCSNHC}_6\text{H}_4\text{Cl-}o$ ,         | m. 198°. <sup>446</sup>          |
| 1,4- $(\text{i-Pr})\text{NHCSNHC}_6\text{H}_4\text{NHCSNHC}_6\text{H}_4\text{Cl-}p$ ,         | m. 191°. <sup>446</sup>          |
| 1,4- $(\text{i-Pr})\text{NHCSNHC}_6\text{H}_4\text{NHCSNHC}_6\text{H}_4\text{Br-}m$ ,         | m. 172°. <sup>446</sup>          |
| 1,4- $(\text{i-Pr})\text{NHCSNHC}_6\text{H}_4\text{NHCSNHC}_6\text{H}_4\text{Br-}p$ ,         | m. 218°. <sup>446</sup>          |
| 1,4- $(\text{i-Pr})\text{NHCSNHC}_6\text{H}_4\text{NHCSNHC}_6\text{H}_4\text{OMe-}o$ ,        | m. 190°. <sup>446</sup>          |
| 1,4- $(\text{i-Pr})\text{NHCSNHC}_6\text{H}_4\text{NHCSNHC}_6\text{H}_4\text{OMe-}p$ ,        | m. 175°. <sup>446</sup>          |
| 1,4- $(\text{i-Pr})\text{NHCSNHC}_6\text{H}_4\text{NHCSNHC}_6\text{H}_4\text{Me-}o$ ,         | m. 191°. <sup>446</sup>          |
| 1,4- $(\text{i-Pr})\text{NHCSNHC}_6\text{H}_4\text{NHCSNHC}_6\text{H}_4\text{Me-}m$ ,         | m. 173°. <sup>446</sup>          |
| 1,4- $(\text{i-Pr})\text{NHCSNHC}_6\text{H}_4\text{NHCSNHC}_6\text{H}_4\text{Me-}p$ ,         | m. 170°. <sup>446</sup>          |
| 1,4-BuNHCSNHC <sub>6</sub> H <sub>4</sub> NHCSNHPh,   | m. 196° and <sup>217°. 446</sup> |
| 1,4-BuNHCSNHC <sub>6</sub> H <sub>4</sub> NHCSNHC <sub>6</sub> H <sub>4</sub> Cl- <i>o</i> ,  | m. 183°. <sup>446</sup>          |
| 1,4-BuNHCSNHC <sub>6</sub> H <sub>4</sub> NHCSNHC <sub>6</sub> H <sub>4</sub> Cl- <i>p</i> ,  | m. 221°. <sup>446</sup>          |
| 1,4-BuNHCSNHC <sub>6</sub> H <sub>4</sub> NHCSNHC <sub>6</sub> H <sub>4</sub> Br- <i>m</i> ,  | m. 180°. <sup>446</sup>          |
| 1,4-BuNHCSNHC <sub>6</sub> H <sub>4</sub> NHCSNHC <sub>6</sub> H <sub>4</sub> Br- <i>p</i> ,  | m. 220°. <sup>446</sup>          |
| 1,4-BuNHCSNHC <sub>6</sub> H <sub>4</sub> NHCSNHC <sub>6</sub> H <sub>4</sub> OMe- <i>o</i> , | m. 182°. <sup>446</sup>          |
| 1,4-BuNHCSNHC <sub>6</sub> H <sub>4</sub> NHCSNHC <sub>6</sub> H <sub>4</sub> OMe- <i>p</i> , | m. 182°. <sup>446</sup>          |
| 1,4-BuNHCSNHC <sub>6</sub> H <sub>4</sub> NHCSNHC <sub>6</sub> H <sub>4</sub> Me- <i>o</i> ,  | m. 187°. <sup>446</sup>          |
| 1,4-BuNHCSNHC <sub>6</sub> H <sub>4</sub> NHCSNHC <sub>6</sub> H <sub>4</sub> Me- <i>m</i> ,  | m. 177°. <sup>446</sup>          |

|   |                         |
|---|-------------------------|
| 1,4-BuNHCSNHC <sub>6</sub> H <sub>4</sub> NHCSNHC <sub>6</sub> H <sub>4</sub> Me- <i>p</i> ,  | m. 196°. <sup>446</sup> |
| 1,2-[PhNHCSNH] <sub>2</sub> C <sub>6</sub> H <sub>4</sub> ,   | m. 176°. <sup>334</sup> |
| 1,3,4-H <sub>2</sub> NCSNHMeC <sub>6</sub> H <sub>3</sub> NHCSNH <sub>2</sub> ,   | m. 285°. <sup>97</sup>  |
| [CH <sub>2</sub> :CHCH <sub>2</sub> NHCSNH] <sub>2</sub> C <sub>6</sub> H <sub>4</sub> - <i>m</i> ,   | m. 102°. <sup>445</sup> |
| [CH <sub>2</sub> :CHCH <sub>2</sub> NHCSNH] <sub>2</sub> C <sub>6</sub> H <sub>4</sub> - <i>p</i> ,   | m. 200°. <sup>445</sup> |
| [ <i>p</i> -C <sub>3</sub> H <sub>5</sub> NHCSNHC <sub>6</sub> H <sub>4</sub> ] <sub>2</sub> SO <sub>2</sub> ,                                | m. 181°. <sup>654</sup> |
| [ <i>p</i> -PhNHCSNHC <sub>6</sub> H <sub>4</sub> ] <sub>2</sub> SO <sub>2</sub> ,  | m. 215°. <sup>654</sup> |
| [ <i>p</i> -( <i>p</i> -ClC <sub>6</sub> H <sub>4</sub> NHCSNHC <sub>6</sub> H <sub>4</sub> )] <sub>2</sub> SO <sub>2</sub> ,                 | m. 198°. <sup>654</sup> |
| [ <i>p</i> -( <i>p</i> -BrC <sub>6</sub> H <sub>4</sub> NHCSNHC <sub>6</sub> H <sub>4</sub> )] <sub>2</sub> SO <sub>2</sub> ,                 | m. 191°. <sup>654</sup> |
| [ <i>p</i> -( <i>p</i> -IC <sub>6</sub> H <sub>4</sub> NHCSNHC <sub>6</sub> H <sub>4</sub> )] <sub>2</sub> SO <sub>2</sub> ,                  | m. 192°. <sup>654</sup> |
| [ <i>p</i> -( <i>o</i> -MeC <sub>6</sub> H <sub>4</sub> NHCSNHC <sub>6</sub> H <sub>4</sub> )] <sub>2</sub> SO <sub>2</sub> ,                 | m. 206°. <sup>654</sup> |
| [ <i>p</i> -( <i>m</i> -MeC <sub>6</sub> H <sub>4</sub> NHCSNHC <sub>6</sub> H <sub>4</sub> )] <sub>2</sub> SO <sub>2</sub> ,                 | m. 210°. <sup>654</sup> |
| [ <i>p</i> -( <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> NHCSNHC <sub>6</sub> H <sub>4</sub> )] <sub>2</sub> SO <sub>2</sub> ,                 | m. 208°. <sup>654</sup> |
| [ <i>o</i> -[ <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> CH <sub>2</sub> NHCSNHC <sub>6</sub> H <sub>4</sub> ]] <sub>2</sub> SO <sub>2</sub> , | m. 211°. <sup>654</sup> |
| [ <i>p</i> -[ <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> CH <sub>2</sub> NHCSNHC <sub>6</sub> H <sub>4</sub> ]] <sub>2</sub> SO <sub>2</sub> , | m. 246°. <sup>654</sup> |
| CH <sub>2</sub> [CH <sub>2</sub> CH <sub>2</sub> SC(:NH)NHNO <sub>2</sub> ] <sub>2</sub> ,  | m. 164°. <sup>404</sup> |
| [·CH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> SC(:NH)NHNO <sub>2</sub> ] <sub>2</sub> ,                   | m. 147°. <sup>404</sup> |
| 1,4-Bis(pseudothiocarbamidomethyl)naphthalene, 2 HCl, m.  | 272°. <sup>43</sup>     |

## POLY-THIOUREAS

|  |                                      |
|--|--------------------------------------|
| Methylene bis(trimethylenethiourea),   | decomposes at 293°. <sup>1237b</sup> |
| Methylene bis(tetramethylenethiourea),   | decomposes at 249°. <sup>1237b</sup> |
| Methylene tris(ethylenethiourea),  | m. 272°. <sup>1237b</sup>            |
| Dimethylene tris(dimethylene thiourea),  | m. 116°. <sup>1237b</sup>            |
| CS(NHCH <sub>2</sub> NHCSNH <sub>2</sub> ) <sub>2</sub> ,  | m. 116°. <sup>1237b</sup>            |
| 1,2,4-(PhNHCSNH) <sub>3</sub> C <sub>6</sub> H <sub>3</sub> ,  | m. 170°. <sup>73</sup>               |
| 1,2,4-( <i>p</i> -BrC <sub>6</sub> H <sub>4</sub> NHCSNH) <sub>3</sub> C <sub>6</sub> H <sub>3</sub> , | m. 183°. <sup>73</sup>               |

## N-CYCLIC THIOUREAS

## RCSR'

| R   | R'   |                           |
|---|--|---------------------------|
| (1,1-Dimethyl-2,2-diphenyl)ethyleneimino, | PhNH,  | m. 94°. <sup>722</sup>    |
| 2-Ethylpyrrolidinyl,                      | PhNH,  | m. 87.6°. <sup>1358</sup> |
| 1-Piperidyl,                              | NH <sub>2</sub> ,                              | m. 128°. <sup>1347</sup>  |
|   | Ph(CH <sub>2</sub> ) <sub>4</sub> NH,          | m. 65°. <sup>134</sup>    |
|   | <i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> NH, | m. 146°. <sup>182</sup>   |

| R  | R'  |   |
|--|---|---|
|  | PhNH,   | m. 99°. <sup>182</sup>  |
|  | <i>p</i> -ClC <sub>6</sub> H <sub>4</sub> NH,         | m. 147°. <sup>173</sup>   |
|  | <i>p</i> -BrC <sub>6</sub> H <sub>4</sub> NH,         | m. 159°. <sup>173</sup>   |
|  | <i>p</i> -FC <sub>6</sub> H <sub>4</sub> NH,          | m. 132°. <sup>173</sup>   |
|  | <i>p</i> -EtOC <sub>6</sub> H <sub>4</sub> NH,        | m. 101°. <sup>173</sup>   |
|  | <i>m</i> -MeC <sub>6</sub> H <sub>4</sub> NH,         | m. 95°. <sup>173</sup>  |
|  | <i>p</i> -EtC <sub>6</sub> H <sub>4</sub> NH,         | m. 109°. <sup>173</sup>   |
|  | 2,4-Me <sub>2</sub> C <sub>6</sub> H <sub>3</sub> NH, | m. 112°. <sup>173</sup>   |
|  | $\alpha$ -C <sub>10</sub> H <sub>7</sub> NH,          | m. 120°. <sup>173</sup>   |
|  | 1-Piperidyl,  | m. 55°. <sup>577</sup> 58°;<br><sup>430</sup> b. 140-60°.<br><sup>577</sup> |
| 1-Piperazyl,                                 | <i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> NH,        | m. 250° dec. <sup>182</sup>   |
| 4-(2-Pyridyl)piperazyl,                      | EtNH,   | m. 99°. <sup>238</sup>  |
|  | CH <sub>2</sub> :CHCH <sub>2</sub> NH,                | m. 89°. <sup>238</sup>  |
|  | PhNH,   | m. 141°. <sup>581</sup>   |
| 4-(2-Pyrimidyl)piperazyl,                    | EtNH,   | m. 135°. <sup>238</sup>   |
|  | PhNH,   | m. 158°. <sup>581</sup>   |
| 4-(2-Pyrazinyl)piperazyl,                    | EtNH,   | m. 148°. <sup>238</sup>   |
|  | CH <sub>2</sub> :CHCH <sub>2</sub> NH,                | m. 103°. <sup>238</sup>   |
|  | PhNH,   | m. 174°. <sup>581</sup>   |
| 4-(2-Thiazolyl)piperazyl,                    | EtNH,   | m. 150°. <sup>238</sup>   |
|  | CH <sub>2</sub> :CHCH <sub>2</sub> NH,                | m. 134°. <sup>238</sup>   |
|  | PhNH,   | m. 181°. <sup>581</sup>   |
| 4-Carbobenzoxyl-1-piperazyl, NH <sub>2</sub> |   | m. 86°. <sup>470</sup>  |
| 4-Morpholinyl,                               | <i>p</i> -ClC <sub>6</sub> H <sub>4</sub> NH,         | m. 172°. <sup>173</sup>   |
|  | <i>p</i> -BrC <sub>6</sub> H <sub>4</sub> NH,         | m. 178°. <sup>173</sup>   |
|  | <i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> NH,        | m. 159°. <sup>173</sup>   |
|  | <i>p</i> -EtOC <sub>6</sub> H <sub>4</sub> NH,        | m. 165°. <sup>173</sup>   |
|  | <i>m</i> -MeC <sub>6</sub> H <sub>4</sub> NH,         | m. 134°. <sup>173</sup>   |
|  | <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> NH,         | m. 150°. <sup>173</sup>   |
|  | <i>p</i> -EtC <sub>6</sub> H <sub>4</sub> NH,         | m. 124°. <sup>173</sup>   |
|  | <i>o</i> -PhC <sub>6</sub> H <sub>4</sub> NH,         | m. 113°. <sup>173</sup>   |
|  | $\alpha$ -C <sub>10</sub> H <sub>7</sub> NH,          | m. 160°. <sup>173</sup>   |
|  | $\beta$ -C <sub>10</sub> H <sub>7</sub> NH,           | m. 174°. <sup>173</sup>   |
| 2-Imino-3-thiazolinyI,                       | C <sub>3</sub> H <sub>5</sub> NH,                     | m. 71°. <sup>432</sup>  |
|  | PhNH,   | m. 60°; <sup>432</sup> pic-<br>rate, m. 230°.<br><sup>432</sup>             |

| R  | R'                                |                        |
|--|-----------------------------------|------------------------|
| 2-Imino-5-methyl-3-oxazolinyI, PhNH,     |                                   | m. 98°. <sup>432</sup> |
| 2-Imino-5-benzylthiomethyl-3-oxazolinyI, |                                   |                        |
|  | C <sub>3</sub> H <sub>5</sub> NH, | m. 87°. <sup>432</sup> |
|  | PhNH,                             | m. 89°. <sup>432</sup> |

## CYCLIC THIOUREAS

|  |  |
|--|--|
| $\overline{\text{NHCH}_2\text{CH}_2\text{NHCS}}$ ,   | m. 174°, <sup>1130</sup> 194°, <sup>438</sup> 195°, <sup>538</sup><br>198°, <sup>6</sup> , <sup>1025</sup> 199°, <sup>1086</sup> |
| $\overline{\text{N}(\text{Bu})\text{CH}_2\text{CH}_2\text{N}(\text{Bu})\text{CS}}$ ,                             | b <sub>8</sub> 185°. <sup>1423</sup>   |
| $\overline{\text{N}(\text{C}_6\text{H}_{11})\text{CH}_2\text{CH}_2\text{N}(\text{C}_6\text{H}_{11})\text{CS}}$ , | m. 226°. <sup>1424</sup>   |
| $\overline{\text{N}(\text{PhCH}_2)\text{CH}_2\text{CH}_2\text{N}(\text{PhCH}_2)\text{CS}}$ ,                     | m. 90°. <sup>828</sup>   |
| $\overline{\text{N}(\text{Ph})\text{CH}_2\text{CH}_2\text{N}(\text{Ph})\text{CS}}$ ,                             | m. 190°. <sup>1423</sup>   |
| $\overline{\text{NHCH}_2\text{CH}_2\text{CH}_2\text{NHCS}}$ ,  | m. 198°. <sup>814</sup>  |
| $\overline{\text{N}(\text{Bu})\text{CH}_2\text{CH}_2\text{CH}_2\text{N}(\text{Bu})\text{CS}}$ ,                  | b <sub>3</sub> 178°. <sup>1423</sup>   |
| $\overline{\text{N}(\text{Me})\text{CH}_2\text{OCH}_2\text{N}(\text{Me})\text{CS}}$ ,                            | m. 112°. <sup>1237b</sup>  |
| $\overline{\text{NHCOCOHNCS}}$ ,   | m. 220°. <sup>100</sup>  |

THIOUREAS FROM HYDROXYLAMINE <sup>736</sup>

|  |                     |
|--|---------------------|
| MeNHCSNHOH,  | decomposes at 101°. |
| MeNHCSNMeOH,   | m. 104°.            |
| MeNHCSNEtOH,   | m. 122°.            |
| EtNHCSNHOH,  | m. 109°.            |
| EtNHCSNEtOH,   | m. 81°.             |
| CH <sub>2</sub> :CHCH <sub>2</sub> NHCSNHOH,                   | decomposes at 120°. |
| CH <sub>2</sub> :CHCH <sub>2</sub> NHCSNMeOH,                  | m. 54°.             |
| CH <sub>2</sub> :CHCH <sub>2</sub> NHCSNEtOH,                  | m. 67°.             |
| CH <sub>2</sub> :CHCH <sub>2</sub> NHCSNPrOH,                  | m. 54°.             |
| CH <sub>2</sub> CHCH <sub>2</sub> NHCSN(PhCH <sub>2</sub> )OH, | m. 58°.             |

## GUANYLTHIOUREAS



| R       |  |
|---------|--|
| Methyl, | m. 159°; <sup>1059</sup> , <sup>1202</sup> carbonate, m. 104°. <sup>1202</sup>                                   |
| Ethyl,  | HCl, m. 158°; <sup>1202</sup> carbonate, m. 94°; <sup>1202</sup><br>nitrate, decomposes at 204°. <sup>1202</sup> |

| R   | R'  |
|---|---|
| Propyl,   | HCl, m. 133°. <sup>1202</sup>   |
| <i>i</i> -Butyl,  | carbonate, decomposes at 91°; <sup>1202</sup> nitrate, m. 179°; <sup>1202</sup> sulfate, m. 120°; <sup>1202</sup> picrate, m. 203°. <sup>1202</sup>       |
| <i>i</i> -Amyl,   | carbonate, m. 82°; <sup>1202</sup> nitrate, m. 176°; <sup>1202</sup> sulfate, m. 96°; <sup>1202</sup> picrate, m. 205°. <sup>1202</sup>                   |
| Allyl,  | carbonate, m. 95°; <sup>1202</sup> nitrate, m. 187°; <sup>1202</sup> sulfate, m. 143°; <sup>1202</sup> picrate, decomposes at 213°. <sup>1202</sup>       |
| Phenyl,   | m. 178°; <sup>1202</sup> HCl, decomposes at 178°; <sup>1202</sup> sulfate decomposes at 282°; <sup>1202</sup> picrate decomposes at 250°. <sup>1202</sup> |
| PhCH <sub>2</sub> CH <sub>2</sub> ,                         | picrate, decomposes at 215°. <sup>1202</sup>  |
| <i>p</i> -ClC <sub>6</sub> H <sub>4</sub> ,                 | m. 161°; <sup>508</sup> 192°. <sup>830</sup>  |
| 3,4-Br(EtO)C <sub>6</sub> H <sub>3</sub> ,                  | HCl, m. 123°. <sup>1202</sup>   |
| <i>p</i> -HOC <sub>6</sub> H <sub>4</sub> ,                 | HCl, m. 199°. <sup>1202</sup>   |
| <i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> ,                | HCl, m. 181°. <sup>1202</sup>   |
| 3,4,5-(MeO) <sub>3</sub> C <sub>6</sub> H <sub>2</sub> ,    | picrate, decomposes at 270°. <sup>1202</sup>  |
| <i>p</i> -EtOC <sub>6</sub> H <sub>4</sub> ,                | m. 148°; <sup>1202</sup> HCl, m. 188°; <sup>1202</sup> formate, m. 164°; <sup>1202</sup> salicylate, decomposes at 139°. <sup>1202</sup>                  |
| <i>p</i> -Me <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> ,  | m. 196°; <sup>1202</sup> sulfate, decomposes at 200°. <sup>1202</sup>   |
| <i>p</i> -EtOCC <sub>6</sub> H <sub>4</sub> ,               | m. 178°; <sup>1202</sup> picrate, decomposes at 250°. <sup>1202</sup>   |
| <i>p</i> -AcOC <sub>6</sub> H <sub>4</sub> ,                | m. 305°. <sup>1202</sup>  |
| <i>m</i> -HOCH <sub>2</sub> C <sub>6</sub> H <sub>4</sub> , | HCl, m. 90°; <sup>1202</sup> picrate, decomposes at 240°. <sup>1202</sup>   |
| PrCO,   | m. 140° dec. <sup>1228b</sup>   |
| C <sub>11</sub> H <sub>23</sub> CO,                         | HCl, m. 156°; <sup>1228b</sup> 157°. <sup>2</sup>   |
| PhCO,   | m. 174°. <sup>1228b</sup>   |



| R                 |                         |
|-------------------|-------------------------|
| Methyl,           | m. 118°. <sup>102</sup> |
| Ethyl,            | m. 98°. <sup>102</sup>  |
| Propyl,           | m. 116°. <sup>102</sup> |
| <i>i</i> -Propyl, | m. 101°. <sup>102</sup> |
| Butyl,            | m. 98°. <sup>102</sup>  |



## OTHER GUANYLTHIOUREAS

|   |  |
|---|--|
| $\text{H}_2\text{NC}(:\text{NH})\text{NHC}(:\text{NH})\text{SEt}$ ,                                     | HBr, m. $90^\circ$ . <sup>621</sup>                                  |
| $\text{H}_2\text{NC}(:\text{NH})\text{NHC}(:\text{NH})\text{SC}_{12}\text{H}_{25}$ ,                    | m. $96^\circ$ ; <sup>621</sup> HBr, m. $140^\circ$ . <sup>1029</sup> |
| $\text{Me}_2\text{NC}(:\text{NMe})\text{NMeCSNMe}_2$ ,  | m. $53^\circ$ . <sup>809</sup>                                       |
| <i>i</i> -PrNHC(:NH)CSNHC <sub>6</sub> H <sub>4</sub> Cl- <i>p</i> ,                                    | m. $139^\circ$ . <sup>506</sup>                                      |
| PhNHC(:NPh)NHCSNHMe,  | m. $166^\circ$ . <sup>1190</sup>                                     |
| PhNHC(:NPh)NHCSNHC <sub>6</sub> H <sub>4</sub> Me- <i>o</i> ,   | m. $119^\circ$ . <sup>1190</sup>                                     |
| <i>o</i> -MeC <sub>6</sub> H <sub>4</sub> NHC[:N( <i>o</i> -MeC <sub>6</sub> H <sub>4</sub> )]NHCSNHPh, | m. $180^\circ$ . <sup>1190</sup>                                     |
| PrNHC(:NH)NHC(:NH)NHCSNHC <sub>6</sub> H <sub>4</sub> Cl- <i>p</i> ,                                    | m. $248^\circ$ . <sup>506</sup>                                      |
| PhNHC(:NH)NHC(:NH)NHCSNHPh,   | m. $248^\circ$ . <sup>506</sup>                                      |
| <i>p</i> -ClC <sub>6</sub> H <sub>4</sub> NHC(:NH)NHC(:NH)NHCSNHPh,                                     | m. $211^\circ$ . <sup>506</sup>                                      |
| (C <sub>3</sub> H <sub>5</sub> NHCSNH) <sub>2</sub> C:NH,   | sinters, $240^\circ$ . <sup>1202</sup>                               |
| ( <i>i</i> -AmNHCSNH) <sub>2</sub> C:NH,  | HCl, m. $100^\circ$ . <sup>1202</sup>                                |
| (PhNHCSNH) <sub>2</sub> C:NH,   | decomposes at $188^\circ$ . <sup>1202</sup>                          |
| ( <i>p</i> -EtOC <sub>6</sub> H <sub>4</sub> NHCSNH) <sub>2</sub> C:NH,                                 | m. $196^\circ$ . <sup>1202</sup>                                     |
| <i>p</i> -C <sub>6</sub> H <sub>4</sub> [NHCSNHC(:NH)NH <sub>2</sub> ] <sub>2</sub> ,                   | 2 HCl, decomposes at $212^\circ$ . <sup>1202</sup>                   |

## MONOTHIABIURETS

|  |                                    |
|--|------------------------------------|
| $\text{H}_2\text{NCSNHCONH}_2$ ,   | m. $186^\circ$ . <sup>541</sup>    |
| $\text{H}_2\text{NCSNHCONHMe}$ ,   | m. $194^\circ$ dec. <sup>541</sup> |
| $\text{H}_2\text{NCSNHCONHEt}$ ,   | m. $184^\circ$ . <sup>541</sup>    |
| $\text{H}_2\text{NCSNHCONHOCCHEt}_2$ ,   | m. $132^\circ$ . <sup>560</sup>    |
| $\text{H}_2\text{NCSNHCONHOCCH(Et)(i-Am)}$ ,   | m. $123^\circ$ . <sup>560</sup>    |
| $\text{H}_2\text{NCSNHCONHOCCH(Et)(CH}_2\text{:CHCH}_2\text{)}$ ,                                    | m. $123^\circ$ . <sup>560</sup>    |
| EtHNCSNHCONHC <sub>6</sub> H <sub>4</sub> Cl- <i>p</i> ,   | m. $184^\circ$ dec. <sup>257</sup> |
| <i>i</i> PrHNCSNHCONHC <sub>6</sub> H <sub>4</sub> Cl- <i>p</i> ,                                    | m. $178^\circ$ . <sup>257</sup>    |
| <i>p</i> -ClC <sub>6</sub> H <sub>4</sub> NHCSNHCONHMe,  | m. $183^\circ$ . <sup>437</sup>    |
| <i>p</i> -ClC <sub>6</sub> H <sub>4</sub> NHCSNHCONHPh,  | m. $165^\circ$ . <sup>437</sup>    |
| <i>p</i> -ClC <sub>6</sub> H <sub>4</sub> NHCSNHC(OMe):NBu,  | m. $96^\circ$ . <sup>257</sup>     |
| <i>p</i> -ClC <sub>6</sub> H <sub>4</sub> NHCSNHC(OMe):NC <sub>6</sub> H <sub>4</sub> Cl- <i>p</i> , | m. $145^\circ$ . <sup>257</sup>    |
| <i>p</i> -ClC <sub>6</sub> H <sub>4</sub> NHCSN:C(OMe)N(Me)( <i>i</i> -Pr),                          | m. $147^\circ$ . <sup>257</sup>    |
| <i>p</i> -ClC <sub>6</sub> H <sub>4</sub> NHCSN:C(OMe)NBu <sub>2</sub> ,                             | m. $126^\circ$ . <sup>257</sup>    |
| <i>p</i> -ClC <sub>6</sub> H <sub>4</sub> NHC(SMe):NCONHC <sub>6</sub> H <sub>4</sub> Cl- <i>p</i> , | m. $140^\circ$ . <sup>257</sup>    |
| <i>p</i> -ClC <sub>6</sub> H <sub>4</sub> NHC(SCH <sub>2</sub> SH):NCONHMe,                          | m. $161^\circ$ . <sup>257</sup>    |
| <i>p</i> -ClC <sub>6</sub> H <sub>4</sub> NHC(SCH <sub>2</sub> SH):NCONHPh,                          | m. $154^\circ$ . <sup>257</sup>    |
| EtNHC(SET):NCONHC <sub>6</sub> H <sub>4</sub> Cl- <i>p</i> ,   | m. $124^\circ$ . <sup>257</sup>    |
| <i>i</i> -PrNHC(SET):NCONHC <sub>6</sub> H <sub>4</sub> Cl- <i>p</i> ,                               | m. $132^\circ$ . <sup>257</sup>    |

## DITHIOBIURETS

|   |  |
|---|--|
| $\text{H}_2\text{NCSNHCSNHMe}$ ,  | m. $153^\circ$ . <sup>541</sup>                              |
| $\text{H}_2\text{NCSNHCSNHEt}$ ,  | m. $175^\circ$ dec. <sup>541</sup>                           |
| $\text{H}_2\text{NCSNHCSNHPr}$ ,  | m. $121^\circ$ . <sup>541</sup>                              |
| $\text{H}_2\text{NCSNHCSNHCH}_2\text{CH:CH}_2$ ,  | m. $138^\circ$ . <sup>541</sup>                              |
| $\text{H}_2\text{NCSNHCSNHPh}$ ,  | m. $174^\circ$ . <sup>541</sup>                              |
| $\text{H}_2\text{NCSNHCSNHC}_6\text{H}_4\text{Cl-}m$ ,  | m. $164^\circ$ . <sup>1304</sup>                             |
| $\text{H}_2\text{NCSNHCSNHC}_6\text{H}_4\text{Cl-}p$ ,  | m. $154^\circ$ . <sup>505</sup> $158^\circ$ . <sup>506</sup> |
| $\text{H}_2\text{NCSNHCSNHC}_6\text{H}_4\text{Br-}p$ ,  | m. $169^\circ$ . <sup>1304</sup>                             |
| $\text{H}_2\text{NCSNHCSNHC}_6\text{H}_4\text{I-}p$ ,   | m. $240^\circ$ . <sup>1304</sup>                             |
| $\text{H}_2\text{NCSNHCSNHC}_6\text{H}_4\text{OMe-}o$ ,   | m. $148^\circ$ . <sup>431</sup>                              |
| $\text{H}_2\text{NCSNHCSNHC}_6\text{H}_4\text{NH}_2\text{-}m$ ,                                     | m. $226^\circ$ . <sup>1304</sup>                             |
| $\text{H}_2\text{NCSNHCSNHC}_6\text{H}_4\text{Me-}m$ ,  | m. $159^\circ$ . <sup>1304</sup>                             |
| $\text{H}_2\text{NCSNHCSNHC}_{10}\text{H}_7\text{-}\alpha$ ,  | m. $236^\circ$ . <sup>1304</sup>                             |
| $\text{H}_2\text{NCSNHCSN}(\text{Me})(p\text{-MeC}_6\text{H}_4)$ ,                                  | m. $236^\circ$ . <sup>1304</sup>                             |
| $\text{MeNHCSNHCSNHC}_6\text{H}_4\text{Cl-}p$ ,   | m. $166^\circ$ . <sup>257</sup>                              |
| $\text{EtNHCSNHCSNHC}_6\text{H}_4\text{Cl-}p$ ,   | m. $160^\circ$ . <sup>257</sup>                              |
| $\text{PrNHCSNHCSNHC}_6\text{H}_4\text{Cl-}p$ ,   | m. $141^\circ$ . <sup>257</sup>                              |
| $i\text{-PrNHCSNHCSNHC}_6\text{H}_4\text{Cl-}m$ ,   | m. $129^\circ$ . <sup>257</sup>                              |
| $i\text{-PrNHCSNHCSNHC}_6\text{H}_4\text{Cl-}p$ ,   | m. $137^\circ$ . <sup>437</sup> $148^\circ$ . <sup>257</sup> |
| $i\text{-PrNHCSNHCSNHC}_6\text{H}_3\text{Cl}_2\text{-}3,4$ ,  | m. $158^\circ$ . <sup>257</sup>                              |
| $i\text{-PrNHCSNHCSNHC}_6\text{H}_4\text{Br-}p$ ,   | m. $152^\circ$ . <sup>257</sup>                              |
| $i\text{-PrNHCSNHCSNHC}_6\text{H}_4\text{I-}p$ ,  | m. $137^\circ$ . <sup>257</sup>                              |
| $p\text{-ClC}_6\text{H}_4\text{NHCSNHCSNHC}_6\text{H}_4\text{Cl-}p$ ,                               | m. $168^\circ$ . <sup>437</sup>                              |
| $p\text{-ClC}_6\text{H}_4\text{NHC}(\text{SMe})\text{:NCSNHC}_6\text{H}_4\text{Cl-}p$ ,             | m. $160^\circ$ . <sup>257</sup>                              |
| $\alpha\text{-C}_{10}\text{H}_7\text{NH}(\text{SMe})\text{:NCSNHC}_{10}\text{H}_7\text{-}\alpha$ ,  | m. $138.5^\circ$ . <sup>70</sup>                             |
| $p\text{-ClC}_6\text{H}_4\text{NHC}(\text{SCH}_2\text{SH})\text{:NHCSNHC}_6\text{H}_4\text{Cl-}p$ , | m. $155^\circ$ . <sup>437</sup>                              |
| $\text{H}_2\text{NC}(\text{SEt})\text{:NCSNHC}_6\text{H}_4\text{Cl-}p$ ,                            | m. $122^\circ$ . <sup>506</sup>                              |
| $\text{MeNHC}(\text{SEt})\text{:NCSNHC}_6\text{H}_4\text{Cl-}p$ ,                                   | m. $105^\circ$ . <sup>257</sup>                              |
| $\text{EtNHC}(\text{SEt})\text{:NCSNHC}_6\text{H}_4\text{Cl-}p$ ,                                   | m. $112^\circ$ . <sup>257</sup>                              |
| $i\text{-PrNHC}(\text{SEt})\text{:NCSNHC}_6\text{H}_4\text{Cl-}m$ ,                                 | m. $81^\circ$ . <sup>257</sup>                               |
| $i\text{-PrNHC}(\text{SEt})\text{:NCSNHC}_6\text{H}_4\text{Cl-}p$ ,                                 | m. $224^\circ$ . <sup>257</sup>                              |
| $i\text{-PrNHC}(\text{SEt})\text{:NCSNHC}_6\text{H}_3\text{Cl}_2\text{-}3,4$ ,                      | m. $104^\circ$ . <sup>257</sup>                              |
| $i\text{-PrNHC}(\text{SEt})\text{:NCSNHC}_6\text{H}_4\text{Br-}p$ ,                                 | m. $130^\circ$ . <sup>257</sup>                              |
| $i\text{-PrNHC}(\text{SEt})\text{:NCSNHC}_6\text{H}_4\text{I-}p$ ,                                  | m. $125^\circ$ . <sup>257</sup>                              |
| $\text{BuNHC}(\text{SEt})\text{:NCSNHC}_6\text{H}_4\text{Cl-}p$ ,                                   | m. $79^\circ$ . <sup>257</sup>                               |
| $p\text{-ClC}_6\text{H}_4\text{NHCS}(\text{SEt})\text{:NCSNH}_2$ ,                                  | m. $163^\circ$ . <sup>506</sup>                              |
| $p\text{-ClC}_6\text{H}_4\text{NHC}(\text{SEt})\text{:NCSNH}(i\text{-Pr})$ ,                        | m. $84^\circ$ . <sup>257</sup>                               |

|   |  |
|---|--|
| $\text{Me}_2\text{NC}(\text{SEt}) : \text{NCSNHC}_6\text{H}_4\text{Cl-}p$ ,                     | m. $174^\circ$ . <sup>257</sup>                                    |
| $(\text{Et})(i\text{-Pr})\text{NC}(\text{SEt}) : \text{NCSNHC}_6\text{H}_4\text{Cl-}p$ ,        | m. $126^\circ$ . <sup>257</sup>                                    |
| $i\text{-PrN} : \text{C}(\text{SEt})\text{NC}(\text{SEt}) : \text{NC}_6\text{H}_4\text{Cl-}p$ , | m. $40^\circ$ ; <sup>257</sup> HI, m. $136^\circ$ . <sup>257</sup> |

## S-SUBSTITUTED THIOUREAS AND THIURONIUM SALTS



R

Methyl,

m.  $79^\circ$ ; <sup>22</sup> sulfate, m.  $244^\circ$  dec.; <sup>22</sup> chloroacetate, decomposes at  $157^\circ$ , <sup>1274</sup>  $162^\circ$ ; <sup>1383</sup> dichloroacetate, decomposes at  $165^\circ$ ; <sup>1274</sup> trichloroacetate decomposes at  $187^\circ$ ; <sup>1274</sup> *p*-toluene-sulfonate, m.  $145^\circ$ .<sup>737</sup>

Ethyl,

m.  $88^\circ$ ; <sup>1383</sup> HBr, m.  $83^\circ$ ; <sup>681</sup> *p*-toluene-sulfonate, m.  $126^\circ$ .<sup>737</sup>

Propyl,

HBr, m.  $60^\circ$ .<sup>1881</sup>

*i*-Propyl,

HBr, m.  $77^\circ$ ; <sup>590a</sup>, <sup>636</sup>  $78^\circ$ ; <sup>675</sup> HI, m.  $69^\circ$ .<sup>636</sup>

Butyl,

HBr, m.  $74^\circ$ ; <sup>681</sup> *p*-toluenesulfonate, m.  $163.5^\circ$ , <sup>681</sup>  $166^\circ$ .<sup>737</sup>

*i*-Butyl,

HBr, m.  $96^\circ$ ; <sup>1381</sup> *p*-toluenesulfonate, m.  $157^\circ$ .<sup>737</sup>

*i*Amyl,

HBr, m.  $84^\circ$ .<sup>1381</sup>

Hexyl,

*p*-toluenesulfonate, m.  $140.5^\circ$ .<sup>737</sup>

Heptyl,

HBr, m.  $94^\circ$ ; <sup>675</sup> acetate, m.  $137^\circ$ .<sup>675</sup>

Octyl,

*p*-toluenesulfonate, m.  $113^\circ$ .<sup>737</sup>

*s*-Octyl,

picrate, m.  $132^\circ$ .<sup>681</sup>

Decyl,

m.  $67^\circ$ ; <sup>1029</sup> HCl, m.  $115^\circ$ ; <sup>56</sup> HBr, m.  $97^\circ$ ; <sup>56</sup>  $99^\circ$ ; <sup>1029</sup> HI, m.  $68^\circ$ .<sup>56</sup>

 $\text{C}_{11}\text{H}_{23}$ ,

picrate, m.  $139^\circ$ .<sup>681</sup>

 $\text{C}_{12}\text{H}_{25}$ ,

m.  $82^\circ$ ; <sup>621</sup>, <sup>1029</sup> HCl, m.  $105^\circ$ , <sup>56</sup>  $133^\circ$ , <sup>621</sup>, <sup>1029</sup>  $135^\circ$ ; <sup>1231</sup> HBr, m.  $91^\circ$ , <sup>56</sup>  $111^\circ$ , <sup>621</sup>, <sup>1029</sup>  $114^\circ$ , <sup>1231</sup> HI, m.  $74^\circ$ ; <sup>56</sup> *p*-toluenesulfonate, m.  $103^\circ$ .<sup>737</sup>

 $\text{C}_{14}\text{H}_{29}$ ,

HCl, m.  $112^\circ$ ; <sup>56</sup> HBr, m.  $106^\circ$ ; <sup>56</sup> HI, m.  $91^\circ$ .<sup>56</sup>

 $\text{C}_{16}\text{H}_{33}$ ,

HCl, m.  $122^\circ$ , <sup>56</sup>  $126^\circ$ ; <sup>1231</sup> HBr, m.  $93^\circ$ , <sup>655</sup>  $105^\circ$ ; <sup>56</sup> HI, m.  $90^\circ$ , <sup>56</sup> picrate, m.  $137^\circ$ .<sup>820</sup>

## R

|  |  |
|--|--|
| $C_{18}H_{37}$ ,   | m. $91^{\circ}$ ; <sup>816</sup> HCl, m. $132^{\circ}$ ; <sup>816</sup> p-toluene-sulfonate, m. $104.5^{\circ}$ . <sup>737</sup>   |
| Oleyl,   | m. $83^{\circ}$ ; <sup>816</sup> HBr, m. $109^{\circ}$ . <sup>816</sup>  |
| Cyclohexyl,  | HBr, m. $200^{\circ}$ , <sup>1235</sup> $207^{\circ}$ ; <sup>42</sup> p-toluenesulfonate, m. $169^{\circ}$ dec. <sup>737</sup>   |
| Phenyl,  | m. $97^{\circ}$ . <sup>21</sup>  |
| $PhCH_2$ ,   | HCl, m. $145^{\circ}$ , <sup>395</sup> $146^{\circ}$ and $172^{\circ}$ , <sup>124</sup> $148^{\circ}$ and $174^{\circ}$ , <sup>192, 317</sup> $150^{\circ}$ , <sup>655</sup> $151^{\circ}$ and $176^{\circ}$ ; <sup>1821</sup> HBr, m. $129^{\circ}$ , <sup>681</sup> $157^{\circ}$ ; <sup>655</sup> HI, m. $139^{\circ}$ ; <sup>681</sup> carbonate, m. $105^{\circ}$ ; <sup>124</sup> perchlorate, m. $182^{\circ}$ ; <sup>681</sup> sulfate, m. $190^{\circ}$ ; <sup>1178</sup> p-toluenesulfonate, m. $182.5^{\circ}$ . <sup>737</sup> |
| p-MeC <sub>6</sub> H <sub>4</sub> CH <sub>2</sub> ,                  | m. $205^{\circ}$ . <sup>910</sup>  |
| $PhCH_2CH_2$ ,   | HCl, m. $160^{\circ}$ ; <sup>306</sup> HBr, m. $186^{\circ}$ ; <sup>558</sup> nitrate, m. $154^{\circ}$ ; <sup>306</sup> picrate, m. $160^{\circ}$ . <sup>306</sup>  |
| L-(1-Phenylethyl),   | HCl, m. $151^{\circ}$ ; <sup>1183</sup> $[\alpha]_D^{22}$ $-47.2^{\circ}$ . <sup>1183</sup>  |
| $PhCH(Me)$ ,   | HBr, m. $159^{\circ}$ ; <sup>1183</sup> picrate, m. $166^{\circ}$ . <sup>1183</sup>  |
| $Ph_2CH$ ,   | HCl, m. $196^{\circ}$ ; <sup>19</sup> HBr, m. $182^{\circ}$ , <sup>120</sup> $186^{\circ}$ .<br>272  |
| $Ph_3C$ ,  | picrate, m. $172^{\circ}$ . <sup>820</sup>   |
| $C_{12}H_{25}OCH_2$ ,  | HCl, m. $134^{\circ}$ . <sup>621</sup>   |
| 4-ClC <sub>6</sub> H <sub>4</sub> OCH <sub>2</sub> ,                 | HCl, m. $154^{\circ}$ . <sup>59</sup>  |
| 2,4-Cl <sub>2</sub> C <sub>6</sub> H <sub>3</sub> OCH <sub>2</sub> , | HCl, m. $165^{\circ}$ ; <sup>59</sup> HI, m. $170^{\circ}$ . <sup>59</sup>   |
| NCCH <sub>2</sub> ,  | HCl, decomposes at $105^{\circ}$ . <sup>907</sup>  |
| ClCH <sub>2</sub> CH <sub>2</sub> ,                                  | p-toluenesulfonate m. $142.5^{\circ}$ . <sup>737</sup>   |
| HOOCCH(Cl)CH <sub>2</sub> ,  | HCl, decomposes at $163^{\circ}$ . <sup>75</sup>   |
| MeOOCCH(Cl)CH <sub>2</sub> ,   | HCl, decomposes at $180^{\circ}$ . <sup>75</sup>   |
| HOOCCH(Br)CH <sub>2</sub> ,  | HCl, decomposes at $145^{\circ}$ . <sup>75</sup>   |
| HOCH <sub>2</sub> CH <sub>2</sub> ,                                  | HCl, m. $111^{\circ}$ ; <sup>973</sup> picrate, m. $156^{\circ}$ . <sup>681</sup>  |
| EtOCH <sub>2</sub> CH <sub>2</sub> ,                                 | HBr, m. $29^{\circ}$ . <sup>973</sup>  |
| Et <sub>2</sub> NCH <sub>2</sub> CH <sub>2</sub> ,                   | b <sub>10</sub> $143^{\circ}$ . <sup>518</sup>   |
| HOOCCH <sub>2</sub> CH <sub>2</sub> ,                                | HCl, m. $145^{\circ}$ . <sup>75</sup>  |
| MeCH(OH)CH <sub>2</sub> ,  | Benzoate, m. $114^{\circ}$ . <sup>115</sup>  |
| NCCH <sub>2</sub> CH <sub>2</sub> ,                                  | HCl, m. $166^{\circ}$ . <sup>907</sup>   |
| HOCH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> ,                  | m. $106^{\circ}$ ; <sup>828</sup> HCl, m. $130^{\circ}$ . <sup>978</sup>   |
| NCCH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> ,                  | HCl, m. $127^{\circ}$ ; <sup>907</sup> picrate, m. $164.5^{\circ}$ . <sup>907</sup>  |
| HOOCCH(Cl)CH(Me),  | HCl, decomposes at $163^{\circ}$ . <sup>75</sup>   |
| HOOCCH <sub>2</sub> CH(Me),  | HCl, m. $167^{\circ}$ . <sup>75</sup>  |

## R

- $\text{Me}_2\text{NCH}_2\text{CH}_2\text{CH}_2\text{CH}_2$ , HCl, m.  $183^\circ$ .<sup>926</sup>  
 $\text{MeC}(\text{Cl})\text{:CHCH}_2$ , m.  $136^\circ$ ; <sup>371</sup> Ac., m.  $130^\circ$ .<sup>371</sup>  
 $\text{HOCH}_2\text{C}(\text{Me})\text{:CH}_2$ , picrate, m.  $183^\circ$ .<sup>975</sup>  
 $\text{MeOCH}_2\text{C}(\text{Me})\text{:CH}_2$ , picrate, m.  $178^\circ$ .<sup>975</sup>  
 $\text{EtOCH}_2\text{C}(\text{Me})\text{:CH}_2$ , picrate, m.  $162^\circ$ .<sup>975</sup>  
 $\text{PrOCH}_2\text{C}(\text{Me})\text{:CH}_2$ , picrate, m.  $137.3^\circ$ .<sup>975</sup>  
*i*- $\text{PrOCH}_2\text{C}(\text{Me})\text{:CH}_2$ , picrate, m.  $139^\circ$ .<sup>975</sup>  
 $\text{BuOCH}_2\text{C}(\text{Me})\text{:CH}_2$ , picrate, m.  $119^\circ$ .<sup>975</sup>  
 $\text{MeOOCCH}_2\text{C}(\text{Me})\text{:CH}_2$ , picrate, m.  $132.5^\circ$ .<sup>975</sup>  
 $\text{EtOOCCH}_2\text{C}(\text{Me})\text{:CH}_2$ , picrate, m.  $141.5^\circ$ .<sup>975</sup>  
 $\text{PrOOCCH}_2\text{C}(\text{Me})\text{:CH}_2$ , picrate, m.  $107.7^\circ$ .<sup>975</sup>  
*i*- $\text{PrOOCCH}_2\text{C}(\text{Me})\text{:CH}_2$ , picrate, m.  $117^\circ$ .<sup>975</sup>  
*trans*-2-Hydroxycyclopentyl, m.  $102^\circ$  dec.; sulfate, m.  $206^\circ$ .<sup>115</sup>  
*trans*-2-Hydroxycyclohexyl, sulfate, m.  $315^\circ$ .<sup>115</sup>  
2,5-(HO)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>, HCl, decomposes at  $160^\circ$ .<sup>164</sup>  
Picryl, picrate, m.  $150^\circ$ .<sup>1275</sup>  
*p*-BrC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>, m.  $127^\circ$  and  $199^\circ$ .<sup>185</sup>  
*o*-MeOC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>, m.  $190^\circ$ .<sup>910</sup>  
*m*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>, m.  $188^\circ$ .<sup>910</sup>  
*p*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>, m.  $197^\circ$ .<sup>910</sup>  
*o*-MeC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>, m.  $200^\circ$ .<sup>910</sup>  
*m*-MeC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>, m.  $177^\circ$ .<sup>910</sup>  
 $\alpha$ -[1,4-(HO)<sub>2</sub>C<sub>10</sub>H<sub>5</sub>], HCl, m.  $218^\circ$  dec.<sup>164</sup>  
 $\beta$ -[1,4-(HO)<sub>2</sub>C<sub>10</sub>H<sub>5</sub>], HCl,  $180^\circ$  dec.<sup>164</sup>  
 $\beta$ -[1,4,3-(HO)<sub>2</sub>MeC<sub>10</sub>H<sub>4</sub>], decomposes at  $250^\circ$ .<sup>164</sup>  
Phthalimidomethyl, HBr, m.  $220^\circ$  dec.<sup>490</sup>  
2-Phthalimidoethyl, HBr, m.  $230^\circ$ .<sup>227</sup>  
3-Phthalimidopropyl, HBr, m.  $231^\circ$ ,<sup>227</sup>  $228^\circ$  dec.<sup>490</sup>  
4-Phthalimidobutyl, HBr, m.  $177^\circ$ .<sup>490</sup>  
5-Phthalimidoamyl, HBr, m.  $189.5^\circ$ .<sup>490</sup>  
Tetraacetyl-D-glucoside, HBr, m.  $192^\circ$ ; <sup>1144</sup>  $[\alpha]_D^{20} -8.72^\circ$ .<sup>1144</sup>  
Tetraacetyl- $\beta$ -D-glucopyranosyl, HBr, m.  $205^\circ$ ; <sup>114</sup>  $[\alpha]_D^{23} -7.6^\circ$ ;  
<sup>114</sup> HAc, m.  $144^\circ$ ; <sup>114</sup>  $[\alpha]_D^{20-23} -10.7^\circ$ .<sup>114</sup>  
Tetraacetyl- $\beta$ -D-galactopyranosyl, HBr, m.  $169.5^\circ$ ; <sup>114</sup>  $[\alpha]_D^{25}$   
 $16.0^\circ$ .<sup>114</sup>  
Triacetyl- $\beta$ -D-xylopyranosyl, HCl, m.  $181^\circ$ ; <sup>114</sup>  $[\alpha]_D^{25} -71.5^\circ$ .<sup>114</sup>  
Heptaacetyl- $\beta$ -D-cellobiosyl, HBr, m.  $179^\circ$ ; <sup>114</sup>  $[\alpha]_D^{25} -26.7^\circ$ .<sup>114</sup>  
2-Pyridyl, HBr, m.  $127^\circ$ .<sup>106</sup>

## R

4-Phenyl-2-thiazolyl, m. 166°; <sup>90</sup> 1,3-diAc., m. 210°. <sup>90</sup>  
 2-Amino-4-thiazolylmethyl, 2HCl, m. 255° dec. <sup>1232</sup>  
 2-Methyl-4-thiazolylmethyl, 2HCl, m. 179.5°. <sup>1232</sup>  
 2 Phenyl-4-thiazolylmethyl, 2HCl, m. 202°. <sup>1232</sup>  
 2,4-Dimethyl-6-pyrimidyl, HCl, m. 153°. <sup>1021</sup>  
 4,6-Dimethyl-2-pyrimidyl, HCl, m. 191° dec. <sup>106</sup>  
 2-Methylmercapto-4-methyl-6-pyrimidyl, HCl, m. 207°. <sup>1021</sup>  
 2-Ethylmercapto-4-methyl-6-pyrimidyl, HCl, m. 204°, <sup>1021</sup> 205°. <sup>1021</sup>  
 4-Amino-5-nitro-6-methyl-2-pyrimidyl, HCl, decomposes at 260°. <sup>12b, 875</sup>  
 2-(1-Pyrrolidyl)ethyl, 2HCl, m. 175°. <sup>755</sup>  
 2-(2-Methyl-1-pyrrolidyl)ethyl, 2HCl, m. 218°. <sup>755</sup>  
 2-(2-Amino-4-methyl-5-(thiazolyl)ethyl, 2HCl, m. 242° dec.; <sup>1270</sup> 2 picrate, m. 233° dec. <sup>1270</sup>  
 MeOOC m. 63.5°. <sup>307</sup>  
 (Me) (Ph) NCO, HCl, m. 175°; <sup>306</sup> nitrate, m. 162°; <sup>306</sup> picrate, m. 175°. <sup>306</sup>  
 (PhCH<sub>2</sub>) (Ph) NCO, nitrate, m. 154°; <sup>306</sup> picrate, m. 161°. <sup>306</sup>  
 Ph<sub>2</sub>NCO, HCl, m. 183°; <sup>306</sup> nitrate, m. 177°. <sup>306</sup>  
 Ac., HCl, m. 100°; <sup>916</sup> picrate, m. 120°. <sup>306</sup>  
 Bz., HCl, m. 116°. <sup>306</sup>  
 BzCH<sub>2</sub>CH<sub>2</sub>, HCl, m. 143° dec. <sup>190</sup>  
 BzCH:CH, HCl, m. 169° dec. <sup>190</sup>

## S AND N-SUBSTITUTED ISOTHIUREAS

RSC(:NR')NR''R'''

| R   | R'             | R''   | R'''   |
|-----|----------------|---|--|
| Me, | H,             | Me,   | PhCH <sub>2</sub> , HI, m. 125°. <sup>401</sup>  |
| Me, | H,             | Ph,   | Ph, HI, m. 160°; <sup>72</sup> picrate, m. 166.5°. <sup>72</sup>   |
| Me, | H,             | <i>p</i> -ClC <sub>6</sub> H <sub>4</sub> , | <i>p</i> -ClC <sub>6</sub> H <sub>4</sub> , m. 200°. <sup>72</sup>   |
| Me, | Me,            | H,  | H, HI, m. 136°. <sup>1121</sup>  |
| Me, | Me,            | Me,   | H, m. 54.5°; <sup>802</sup> b <sub>14</sub> 90°; <sup>802</sup> HI, m. 212°. <sup>1121</sup>                             |
| Me, | Me,            | Me,   | Me, b. 176°; <sup>281</sup> d 0/4 1.0194, <sup>281</sup> d 16/4 1.0061; <sup>281</sup> picrate, m. 99.5°. <sup>281</sup> |
| Me, | Me,            | Et,   | Et, b <sub>10</sub> 80°. <sup>805</sup>  |
| Me, | Me,            | Ph,   | H, HI, m. 157.5°. <sup>401</sup>   |
| Me, | Me,            | PhCH <sub>2</sub> ,                         | H, HI, m. 110.5°. <sup>401</sup>   |
| Me, | Et,            | Me,   | Me, b <sub>13</sub> 80°; <sup>802</sup> HI, m. 95°; <sup>802</sup> picrate, m. 83°. <sup>802</sup>                       |
| Me, | Et,            | Me,   | Et, b <sub>11</sub> 80°. <sup>802</sup>  |
| Me, | Et,            | Et,   | H, HI, m. 76.5°; <sup>401</sup> picrate, m. 116°. <sup>957</sup>   |
| Me, | Et,            | Et,   | Et, b <sub>12</sub> 85°. <sup>803</sup>  |
| Me, | <i>i</i> -Pr,  | 6-Methyl-8-quinolyl,                        | H, m. 83.5°. <sup>887</sup>  |
| Me, | Allyl,         | H,  | H, HI, m. 69.5°. <sup>401</sup>  |
| Me, | <i>c</i> -Hex, | H,  | H, HI, m. 144°. <sup>401</sup>   |

| R                   | R'   | R''  | R'''   |
|---------------------|--|--|--|
| Me,                 | <i>c</i> -Hex,                               | PhCH <sub>2</sub> ,                          | H, HI, m. 173.5°. <sup>401</sup>   |
| Me,                 | <i>c</i> -Hex,                               | Ph,  | H, HI, m. 187°. <sup>401</sup>   |
| Me,                 | Ph,  | H,   | H, HI, m. 146°. <sup>401</sup>   |
| Me,                 | Ph,  | Me,  | Me, HI, m. 133.5°. <sup>401</sup>  |
| Me,                 | Ph,  | Ph,  | H, m. 109°. <sup>70</sup>  |
| Me,                 | Ph,  | <i>p</i> -BrC <sub>6</sub> H <sub>4</sub> ,  | H, m. 79°; <sup>265</sup> HI, m. 152°. <sup>265</sup>  |
| Me,                 | <i>p</i> -ClC <sub>6</sub> H <sub>4</sub> ,  | <i>p</i> -ClC <sub>6</sub> H <sub>4</sub> ,  | -H, m. 133°. <sup>292</sup>  |
| Me,                 | <i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> , | H,   | H, HI, m. 163°. <sup>401</sup>   |
| Me,                 | <i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> , | <i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> , | H, m. 85°. <sup>292</sup>  |
| Me,                 | $\alpha$ -C <sub>10</sub> H <sub>7</sub> ,   | H,   | H, m. 89°; <sup>70</sup> HI, m. 183°. <sup>70</sup>  |
| Me,                 | 6-QuinolyI,                                  | NC,  | H, m. 206°. <sup>381</sup>   |
| Me,                 | O <sub>2</sub> N,                            | H,   | H, m. 164°; <sup>404</sup> n <sub>D</sub> <sup>25</sup> 1.486; <sup>404</sup> d <sub>25</sub> 1.93. <sup>404</sup> |
| Me,                 | NC,  | H,   | H, m. 175°. <sup>685</sup>   |
| HSCH <sub>2</sub> , | <i>p</i> -ClC <sub>6</sub> H <sub>4</sub> ,  | H,   | H, HI, m. 167°. <sup>437</sup>   |
| Et,                 | H,   | Me,  | Me, HBr, m. 143°. <sup>257</sup>   |
| Et,                 | H,   | Ph,  | Ph, HI, m. 26°. <sup>72</sup>  |
| Et,                 | H,   | <i>p</i> -ClC <sub>6</sub> H <sub>4</sub> ,  | <i>p</i> -ClC <sub>6</sub> H <sub>4</sub> , m. 134.5°. <sup>72</sup>   |
| Et,                 | H,   | F,   | Ph, HBr, m. 170°. <sup>72</sup>  |
| Et,                 | Me,  | Me,  | Ph, b <sub>10</sub> 111°. <sup>514</sup>   |
| Et,                 | Et,  | Et,  | H, picrate, m. 72°. <sup>957</sup>   |
| Et,                 | Et,  | Et,  | Et, b <sub>15</sub> 110°; <sup>281</sup> b. 216°; <sup>281</sup> d 0/4 0.9426, <sup>281</sup>                      |
|                     |  |  | d 21/4 0.9252. <sup>281</sup>  |
| Et,                 | Ph,  | H,   | H, picrate, m. 199.5°. <sup>737</sup>  |



S AND N-SUBSTITUTED ISOTHIUREAS (*Continued*)

| R                           | R'  | R''   | R'''  |  |
|-----------------------------|---|---|---|--|
| Et,                         | <i>p</i> -BuOC <sub>6</sub> H <sub>4</sub> ,                | H,  | H,  | picrate, m. 163°. <sup>737</sup>             |
| Et,                         | Amidino,  | H,  | H,  | HBr, m. 128°. <sup>780a</sup>                |
| Et,                         | O <sub>2</sub> N,   | H,  | H,  | m. 111°. <sup>404</sup>                      |
| Et,                         | NC,   | H,  | H,  | m. 87°. <sup>685</sup>                       |
| Et,                         | Bz.,  | Bz.,  | H,  | m. 112°. <sup>1148</sup>                     |
| Et,                         | <i>p</i> -O <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> CO, | Et,   | <i>p</i> -O <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> CO,                     | m. 216°. <sup>1148</sup>                     |
| HOOCCH(Cl)CH <sub>2</sub> , | Et,   | H,  | H, HCl, decomposes at 115°. <sup>75</sup>                                       |  |
| Pr,                         | Et,   | Et,   | H,  | picrate, m. 66°. <sup>957</sup>              |
| Pr,                         | Pr,   | Pr,   | Pr, b <sub>15</sub> 154°, <sup>281</sup> b. 270°; <sup>281</sup> d. o/4 0.9179, | <sup>281</sup> d 21/4 0.9014. <sup>281</sup> |
| Pr,                         | Ph,   | Ph,   | H,  | HI, m. 103°. <sup>265</sup>                  |
| Pr,                         | Ph,   | <i>p</i> -BrC <sub>6</sub> H <sub>4</sub> ,         | H,  | m. 84°. <sup>265</sup>                       |
| Pr,                         | <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> ,                 | <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> ,         | H, m. 99°; <sup>265</sup> HI, m. 165°. <sup>265</sup>                           |  |
| Pr,                         | 2,4-Me <sub>2</sub> C <sub>6</sub> H <sub>3</sub> ,         | 2,4-Me <sub>2</sub> C <sub>6</sub> H <sub>3</sub> , | H,  | m. 113.5°. <sup>265</sup>                    |
| Pr,                         | O <sub>2</sub> N,   | H,  | H,  | m. 99°. <sup>404</sup>                       |
| Bu,                         | Ph,   | H,  | H, HI, m. 110°; <sup>737</sup> picrate, m. 144°. <sup>787</sup>                 |  |
| Bu,                         | Ph,   | Ph,   | H,  | HI, m. 122°. <sup>265</sup>                  |
| Bu,                         | <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> ,                 | <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> ,         | H,  | HI, m. 145°. <sup>265</sup>                  |
| Bu,                         | O <sub>2</sub> N,   | H,  | H,  | m. 90.5°. <sup>404</sup>                     |
| Bu,                         | NC,   | H,  | H,  | m. 91°. <sup>685</sup>                       |
| Hex,                        | Me,   | Me,   | Me,   | b <sub>1.0</sub> 95°. <sup>514</sup>         |

| R                                 | R'   | R''           | R'''   |
|-----------------------------------|--|---------------|--|
| Oct,                              | Me,  | Me,           | Me, $b_{0.6} \ 112^\circ$ . <sup>514</sup>   |
| Oct,                              | Ph,  | H,            | H, HBr, m. $96.5^\circ$ ; <sup>737</sup> picrate, m. $130.5^\circ$ . <sup>737</sup>                        |
| Dec,                              | Me,  | Me,           | H, HCl m. $37^\circ$ ; <sup>56</sup> HBr, m. $49^\circ$ ; <sup>56</sup> HI, m. $57^\circ$ . <sup>56</sup>  |
| C <sub>12</sub> H <sub>25</sub> , | Me,  | Me,           | H, HCl, m. $35^\circ$ ; <sup>56</sup> HBr, m. $57^\circ$ ; <sup>56</sup> HI, m. $67^\circ$ . <sup>56</sup> |
| C <sub>14</sub> H <sub>29</sub> , | Me,  | Me,           | H, HCl, m. $39^\circ$ ; <sup>56</sup> HBr, m. $59^\circ$ ; <sup>56</sup> HI, m. $69^\circ$ . <sup>56</sup> |
| C <sub>14</sub> H <sub>29</sub> , | Et,  | Et,           | H, HCl, m. $58^\circ$ ; <sup>56</sup> HBr, m. $53^\circ$ ; <sup>56</sup> HI, m. $39^\circ$ . <sup>56</sup> |
| C <sub>14</sub> H <sub>29</sub> , | <i>i</i> -Pr,                                | <i>i</i> -Pr, | H, HI, m. $41^\circ$ . <sup>56</sup>   |
| C <sub>14</sub> H <sub>29</sub> , | Bu,  | Bu,           | H, HBr, m. $34^\circ$ . <sup>56</sup>  |
| C <sub>16</sub> H <sub>33</sub> , | Me,  | Me,           | H, HCl, m. $54^\circ$ ; <sup>56</sup> HBr, m. $62^\circ$ ; <sup>56</sup> HI, m. $78^\circ$ . <sup>56</sup> |
| C <sub>16</sub> H <sub>33</sub> , | Et,  | Et,           | H, HCl, m. $65^\circ$ ; <sup>56</sup> HBr, m. $56^\circ$ ; <sup>56</sup> HI, m. $45^\circ$ . <sup>56</sup> |
| C <sub>16</sub> H <sub>33</sub> , | <i>i</i> -Pr,                                | <i>i</i> -Pr, | H, HI, m. $51^\circ$ . <sup>56</sup>   |
| C <sub>16</sub> H <sub>33</sub> , | Bu,  | Bu,           | H, HCl, m. $38^\circ$ ; <sup>56</sup> HBr, m. $39^\circ$ ; <sup>56</sup> HI, m. $45^\circ$ . <sup>56</sup> |
| Propylene,                        | Allyl,                                       | H,            | H, m. $56^\circ$ ; <sup>568</sup> picrate, m. $130^\circ$ . <sup>568</sup>                                 |
| Allyl,                            | Me,  | H,            | H, HI, m. $73^\circ$ . <sup>472</sup>  |
| Allyl,                            | Et,  | H,            | H, HBr, m. $74^\circ$ ; <sup>472</sup> HI, m. $72^\circ$ . <sup>472</sup>                                  |
| Allyl,                            | <i>p</i> -EtOC <sub>6</sub> H <sub>4</sub> , | H,            | H, m. $100.5^\circ$ . <sup>895</sup>   |

## S AND N-SUBSTITUTED ISOTHIUREAS (Continued)

| R   | R'   | R''   | R'''                            |                                  |
|---|--|---|---------------------------------|----------------------------------|
| Allyl,  | NC,  | H,  | H,                              | m. 95°. <sup>685</sup>           |
| PhCH <sub>2</sub> ,                                   | Ph,  | H,  | H,                              | m. 80°. <sup>306</sup>           |
| Ph,   | Me,  | H,  | H,                              | HI, m. 147°. <sup>472</sup>      |
| Ph,   | Et,  | H,  | H, HBr, m. 110°; <sup>472</sup> | HI, m. 103°. <sup>472</sup>      |
| Ph,   | <i>p</i> -EtOC <sub>6</sub> H <sub>4</sub> , | H,  | H,                              | m. 129°. <sup>895</sup>          |
| 2,5-(HO) <sub>2</sub> C <sub>6</sub> H <sub>3</sub> , | $\alpha$ -C <sub>10</sub> H <sub>7</sub> ,   | H,  | H,                              | HCl, m. 140°. <sup>472</sup>     |
| 2,5-(HO) <sub>2</sub> C <sub>6</sub> H <sub>3</sub> , | $\beta$ -C <sub>10</sub> H <sub>7</sub> ,    | H,  | H,                              | HCl, m. ca. 200°. <sup>164</sup> |
| Ac.,  | CH <sub>2</sub> :CHCH <sub>2</sub> ,         | H,  | H,                              | HCl, m. 104°. <sup>308a</sup>    |
| Ac.,  | Ph,  | H,  | H, HCl, m. 94°; <sup>306</sup>  | picrate, m. 188°. <sup>306</sup> |
| Ac.,  | Ph,  | Ph,   | H,                              | HCl, m. 106°. <sup>306</sup>     |
| Ac.,  | <i>o</i> -MeC <sub>6</sub> H <sub>4</sub> ,  | H,  | H,                              | HCl, m. 96°. <sup>306</sup>      |
| Ac.,  | <i>o</i> -MeC <sub>6</sub> H <sub>4</sub> ,  | <i>o</i> -MeC <sub>6</sub> H <sub>4</sub> , | H,                              | HCl, m. 136°. <sup>306</sup>     |
| Ac.,  | <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> ,  | H,  | H,                              | HCl, m. 103°. <sup>306</sup>     |
| Bz.,  | Ph,  | Ph,   | H,                              | HCl, m. 109°. <sup>306</sup>     |
| Bz.,  | <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> ,  | H,  | H,                              | HCl, m. 138°. <sup>306</sup>     |
| Bz.,  | <i>p</i> -Morpholinophenyl,                  | H,  | H,                              | m. 185°. <sup>1356</sup>         |
| BzCH:CH <sub>2</sub> ,                                | PhCH <sub>2</sub> ,                          | H,  | H,                              | HCl, m. 154.5°. <sup>190</sup>   |
| Picryl,   | Ph,  | H,  | H,                              | picrate, m. 82°. <sup>1275</sup> |

## DI(S-SUBSTITUTED) ISOTHIURONIUM SALTS



| R  | X  |
|--|--|
| -CH <sub>2</sub> -,  | picrate, m. 232°. <sup>681</sup>   |
| -CH <sub>2</sub> CH <sub>2</sub> -,                                      | chloride, m. 248°; <sup>675</sup> bromide, m. 237°; <sup>494</sup><br>picrate, m. 267° dec. <sup>681</sup> p-toluenesulfonate,<br>m. 279° dec. <sup>737</sup>  |
| -CH(Me)CH <sub>2</sub> -,  | picrate, m. 200°; <sup>566</sup> 232°. <sup>820</sup>  |
| -(CH <sub>2</sub> ) <sub>3</sub> -,                                      | bromide, m. 199.5°; <sup>490</sup> 204°; <sup>494</sup> 229°; <sup>820</sup> pic-<br>rate, m. 128°. <sup>566</sup>   |
| -(CH <sub>2</sub> ) <sub>4</sub> -,                                      | chloride, m. 227.5°; <sup>494</sup> bromide, m. 210°. <sup>303</sup> ,<br><sup>490</sup>   |
| -(CH <sub>2</sub> ) <sub>5</sub> -,                                      | chloride, m. 210°; <sup>699</sup> 213°; <sup>494</sup> chloride, H <sub>2</sub> O,<br>m. 206°; <sup>699</sup> bromide, m. 155°; <sup>303</sup> 161.5°; <sup>490</sup><br>nitrate, m. 119°; <sup>789</sup> picrate, m. 247° dec. <sup>681</sup> |
| -(CH <sub>2</sub> ) <sub>6</sub> -,                                      | bromide, m. 200°; <sup>303</sup> 211°; <sup>494</sup> 231° dec. <sup>699</sup><br>p-toluenesulfonate, m. 236.5°. <sup>737</sup>  |
| -(CH <sub>2</sub> ) <sub>7</sub> -,                                      | bromide, m. 161°; <sup>303</sup> 168°; <sup>494</sup> picrate, m.<br>208°. <sup>681</sup>  |
| -(CH <sub>2</sub> ) <sub>8</sub> -,                                      | chloride, m. 186°; <sup>699</sup> bromide, m. 185°; <sup>303</sup><br>190°; <sup>494</sup> picrate, m. 214°. <sup>681</sup>  |
| -(CH <sub>2</sub> ) <sub>9</sub> -,                                      | bromide, m. 144°; <sup>494</sup> picrate, m. 193°. <sup>681</sup>  |
| -(CH <sub>2</sub> ) <sub>10</sub> -,                                     | chloride, m. 186°; <sup>699</sup> 188°; <sup>494</sup> bromide, m.<br>158°; <sup>303</sup> 160.5°. <sup>490</sup>  |
| -(CH <sub>2</sub> ) <sub>12</sub> -,                                     | bromide, m. 170°. <sup>303</sup>   |
| -(CH <sub>2</sub> C:CCH <sub>2</sub> -,                                  | chloride, m. 183°. <sup>225</sup>  |
| -CH <sub>2</sub> C(:CH <sub>2</sub> )CH <sub>2</sub> -,                  | chloride, m. 140°. <sup>225</sup>  |
| -CH <sub>2</sub> CH(CH:CH <sub>2</sub> )-,                               | chloride, m. 204°. <sup>225</sup>  |
| -CH <sub>2</sub> CH:CClCH <sub>2</sub> -,                                | bromide, m. 205°. <sup>225</sup>   |
| -CH <sub>2</sub> CH:CHCH(Me)-,   | bromide, m. 211°. <sup>225</sup>   |
| -CH <sub>2</sub> CH <sub>2</sub> CH:CHCH <sub>2</sub> CH <sub>2</sub> -, | bromide, m. 174°. <sup>225</sup>   |
| -CH <sub>2</sub> C:CC:CCH <sub>2</sub> -,                                | bromide, m. 67°. <sup>225</sup>  |
| -CH <sub>2</sub> CH:CHCH:CHCH <sub>2</sub> -,                            | bromide, m. 200°. <sup>225</sup>   |
| -CH <sub>2</sub> CH <sub>2</sub> C:CCH <sub>2</sub> CH <sub>2</sub> -,   | bromide, m. 148°. <sup>225</sup>   |
| 2-Amino-4,6-pyrimidylene,  | chloride, decomposes at 200°. <sup>1021</sup>  |
| 6-Methyl-2,4-pyrimidylene,   | chloride, decomposes at 285°. <sup>1021</sup>  |
| 4-Methyl-5-nitro-2,4-pyrimidylene,                                       | chloride, dec. at 290°. <sup>1021</sup>  |

## COMMON ISOTHIURONIUM SALTS USED FOR IDENTIFICATION



| R                 | Picrate   | 3,5-Dinitrobenzoate                          | Styphnate  | Picrolonate             |
|-------------------|---|--|--|-------------------------|
| Methyl,           | m. 224°. <sup>150, 820, 1149</sup>  | m. 205°, <sup>681</sup> 206°. <sup>681</sup> | m. 223° dec., <sup>681</sup> 226° dec. <sup>1149</sup> | m. 237°. <sup>681</sup> |
| Ethyl,            | m. 188°. <sup>150, 820, 1149</sup>  | m. 188°. <sup>681</sup>                      | m. 177° dec. <sup>681</sup> 179°. <sup>1149</sup>      | m. 211°. <sup>681</sup> |
| Propyl,           | m. 177°, <sup>820, 1149</sup> 181°. <sup>150</sup>                            | m. 176°. <sup>681</sup>                      | m. 155.5° dec., <sup>681</sup> 161°. <sup>1149</sup>   | m. 197°. <sup>681</sup> |
| <i>i</i> -Propyl, | m. 190°, <sup>636, 1029</sup> 196°, <sup>820, 1149</sup> 148°. <sup>150</sup> | m. 194°. <sup>681</sup>                      | m. 183°, <sup>681</sup> 187°. <sup>1149</sup>          | m. 209°. <sup>681</sup> |
| Butyl,            | m. 177°, <sup>820, 1149</sup> 180°. <sup>150</sup>                            | m. 169°. <sup>681</sup>                      | m. 159°, <sup>681</sup> 164°. <sup>1149</sup>          | m. 198°. <sup>681</sup> |
| <i>i</i> -Butyl,  | m. 167°, <sup>820, 1149</sup> 174°. <sup>150</sup>                            | m. 158°. <sup>681</sup>                      | m. 143.5°, <sup>681</sup> 150°. <sup>1149</sup>        | m. 219°. <sup>681</sup> |
| <i>s</i> -Butyl,  | m. 166°, <sup>820, 1149</sup> 190°. <sup>150</sup>                            | m. 176°. <sup>681</sup>                      | m. 163°. <sup>1149</sup>                               |                         |
| <i>t</i> -Butyl,  | m. 148° dec. <sup>1149</sup>  |  | m. 151°. <sup>1149</sup>                               |                         |
| Amyl,             | m. 154°, <sup>150, 820</sup> 155°. <sup>1149</sup>                            | m. 156°. <sup>681</sup>                      | m. 141°, <sup>681</sup> 147°. <sup>1149</sup>          | m. 189°. <sup>681</sup> |
| <i>i</i> -Amyl,   | m. 173°, <sup>820, 1149</sup> 179°. <sup>150</sup>                            |  | m. 159°. <sup>1149</sup>                               |                         |
| <i>s</i> -Amyl,   | m. 154°, <sup>150</sup> 155°, <sup>1149</sup> 157°. <sup>820</sup>            |  | m. 112°. <sup>1149</sup>                               |                         |
| <i>t</i> -Amyl,   | m. 127° dec. <sup>1149</sup>  |  | m. 124° dec. <sup>1149</sup>                           |                         |
| 1-Ethylpropyl,    | m. 159°. <sup>1149</sup>  |  | m. 114°. <sup>1149</sup>                               |                         |
| Hexyl,            | m. 157°. <sup>150, 820, 1149</sup>  |  | m. 153°. <sup>1149</sup>                               |                         |
| <i>i</i> -Hexyl,  | m. 147°. <sup>1149</sup>  |  | m. 144°. <sup>1149</sup>                               |                         |
| Heptyl,           | m. 142°, <sup>820</sup> 143°. <sup>1149</sup>                                 | m. 157°, <sup>681</sup> 158°. <sup>681</sup> | m. 144°. <sup>1149</sup>                               | m. 188°. <sup>681</sup> |
| Octyl,            | m. 134°, <sup>820, 1149</sup> 136°. <sup>681</sup>                            | m. 152°. <sup>681</sup>                      | m. 122°. <sup>1149</sup>                               |                         |

| R   | Picrate                                       | 3,5-Dinitrobenzoate                          | Styphnate                                     | Picrolonate               |
|---|---|--|---|---------------------------|
| 2-Ethylhexyl,   | m. 136°. <sup>681</sup>                       | m. 135°, <sup>681</sup> 136°. <sup>681</sup> | m. 198°. <sup>681</sup>                       |                           |
| Nonyl,  | m. 131°. <sup>681</sup>                       | m. 144°. <sup>681</sup>                      |   |                           |
| Decyl,  | m. 137°. <sup>681</sup>                       | m. 145°, <sup>681</sup> 146°. <sup>681</sup> |   |                           |
| C <sub>12</sub> H <sub>25</sub> ,                               | m. 139°. <sup>681</sup>                       | m. 143°. <sup>681</sup>                      |   |                           |
| CH <sub>2</sub> :CHCH <sub>2</sub> ,                            | m. 155°. <sup>150, 820, 1149</sup>            | m. 163°. <sup>681</sup>                      | m. 149°, <sup>681</sup> 154°. <sup>1149</sup> | m. 199.5°. <sup>681</sup> |
| PhCH <sub>2</sub> ,   | m. 187°, <sup>1149</sup> 188°. <sup>681</sup> | m. 175°. <sup>681</sup>                      | m. 186°, <sup>681</sup> 190°. <sup>1149</sup> | m. 202°. <sup>681</sup>   |
| 4-O <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> CH <sub>2</sub> | m. 205°. <sup>1149</sup>                      |  | m. 166°. <sup>1149</sup>                      |                           |

S-BENZYL, S-*p*-CHLOROBENZYL, AND S-*p*-BROMOBENZYL  
ISOTHIURONIUM SALTS FOR THE IDENTIFICATION OF ACIDS

| Acid                          | S-Benzyl  | S- <i>p</i> -Chlorobenzyl   | S- <i>p</i> -Bromobenzyl                    |
|-------------------------------|---|-----------------------------|---|
| Formic,                       | m. 146° <sup>.317</sup> 151° <sup>.1321</sup> 152° <sup>.81</sup>                       | m. 148° <sup>.81, 294</sup> | m. 148° <sup>.81, 293</sup>                 |
| Acetic,                       | m. 134° <sup>.317</sup> 135° <sup>.1264</sup> 136° <sup>.81, 1321</sup>                 | m. 140° <sup>.81, 294</sup> | m. 149° <sup>.81, 293</sup>                 |
| Chloroacetic,                 | m. 160° <sup>.81</sup>  | m. 158° <sup>.81, 294</sup> | m. 154° <sup>.81, 293</sup>                 |
| Propionic,                    | m. 148° <sup>.317</sup> 152° <sup>.1321</sup> 153° <sup>.81</sup> 154° <sup>.1264</sup> | m. 143° <sup>.81, 294</sup> | m. 146° <sup>.81, 293</sup>                 |
| Butyric,                      | m. 146° <sup>.317</sup> 150° <sup>.81</sup>   | m. 139° <sup>.81, 294</sup> | m. 142° <sup>.81, 293</sup>                 |
| Valeric,                      | m. 153° <sup>.573</sup> 156° <sup>.81</sup>   | m. 142° <sup>.81, 294</sup> | m. 146° <sup>.81, 293</sup>                 |
| Isovaleric,                   | m. 153° <sup>.317</sup> 159° <sup>.81</sup>   | m. 148° <sup>.81</sup>      | m. 138° <sup>.81</sup> 148° <sup>.293</sup> |
| Caproic,                      | m. 157° <sup>.81</sup>  | m. 143° <sup>.81, 294</sup> | m. 146° <sup>.81, 293</sup>                 |
| Palmitic,                     | m. 141° <sup>.317, 573</sup>  | m. 146° <sup>.294</sup>     | m. 135° <sup>.293</sup>                     |
| Oleic,                        | m. 134.5° <sup>.696</sup>   | m. 131° <sup>.294</sup>     | m. 133° <sup>.293</sup>                     |
| Benzoic,                      | m. 166° <sup>.317, 573</sup>  | m. 155° <sup>.294</sup>     | m. 154° <sup>.293</sup>                     |
| Salicylic,                    | m. 146° <sup>.317</sup>   | m. 162° <sup>.294</sup>     | m. 168° <sup>.293</sup>                     |
| <i>o</i> -Toluic,             | m. 140° <sup>.317</sup>   | m. 150° <sup>.294</sup>     | m. 151° <sup>.293</sup>                     |
| <i>m</i> -Toluic,             | m. 164° <sup>.317</sup>   | m. 151° <sup>.294</sup>     | m. 151° <sup>.293</sup>                     |
| <i>p</i> -Toluic,             | m. 190° <sup>.317</sup>   | m. 161° <sup>.294</sup>     | m. 165° <sup>.293</sup>                     |
| Cinnamic,                     | m. 175° <sup>.317, 573</sup>  | m. 167° <sup>.294</sup>     | m. 170° <sup>.293</sup>                     |
| Phthalic,                     | m. 151° <sup>.317</sup> 152° <sup>.124</sup>  | m. 166° <sup>.294</sup>     | m. 166° <sup>.293</sup>                     |
| Oxalic,                       | m. 193° <sup>.317</sup> 196° <sup>.1321</sup> 198° <sup>.81</sup> 220° <sup>.124</sup>  | m. 194° <sup>.81, 294</sup> | m. 194° <sup>.81, 293</sup>                 |
| Succinic (2H <sub>2</sub> O), | m. 149° <sup>.317</sup> 153° <sup>.573</sup> 154° <sup>.81</sup> 176° <sup>.124</sup>   | m. 167° <sup>.81, 294</sup> | m. 167° <sup>.81, 293</sup>                 |

## Acid

|                           |   |
|---------------------------|---|
| Beomoacetic,              | benzyl, m. 145°. <sup>427</sup>   |
| Glycolic,                 | benzyl, m. 141°. <sup>317</sup>   |
| Phenoxyacetic,            | benzyl, m. 181°. <sup>427</sup>   |
| Phenylacetic,             | benzyl, m. 166°. <sup>427</sup>   |
| Mandelic,                 | benzyl, m. 166°. <sup>317</sup>   |
| Diphenylacetic,           | benzyl, m. 145°. <sup>317</sup>   |
| Benzilic,                 | benzyl, m. 140°. <sup>427</sup>   |
| Triphenylacetic,          | benzyl, m. 177°. <sup>427</sup>   |
| $\alpha$ -Naphthylacetic, | benzyl, m. 163°. <sup>427</sup>   |
| Hippuric,                 | benzyl, m. 159°. <sup>427</sup>   |
| $\beta$ -Chloropropionic, | benzyl, m. 146°. <sup>1146</sup>  |
| $\beta$ -Bromopropionic,  | benzyl, m. 136°. <sup>1146</sup>  |
| Lactic,                   | benzyl, m. 153°. <sup>317</sup>   |
| Levulinic,                | benzyl, m. 134°. <sup>427</sup>   |
| Pyruvic,                  | benzyl, m. 158°. <sup>427</sup>   |
| Isobutyric,               | benzyl, m. 143°. <sup>317</sup> 150°. <sup>81</sup>                               |
| Heptoic,                  | <i>p</i> -bromobenzyl, m. 147°. <sup>293</sup>                                    |
| Caprylic,                 | benzyl, m. 157°; <sup>81</sup> <i>p</i> -bromobenzyl, m. 147°. <sup>81, 293</sup> |
| Capric,                   | benzyl, m. 149°; <sup>427</sup> <i>p</i> -bromobenzyl, m. 145°. <sup>293</sup>    |
| Lauric,                   | benzyl, m. 141°; <sup>317</sup> <i>p</i> -bromobenzyl, m. 142°. <sup>293</sup>    |
| Myristic,                 | benzyl, m. 139°. <sup>317</sup>   |
| Stearic,                  | benzyl, m. 143°; <sup>317</sup> <i>p</i> -bromobenzyl, m. 135°. <sup>293</sup>    |
| Cyclohexanecarboxylic,    | benzyl, m. 166°. <sup>427</sup>   |
| Acrylic,                  | benzyl, m. 148°. <sup>1146</sup>  |
| Methacrylic,              | benzyl, m. 156°. <sup>427</sup>   |
| Crotonic,                 | benzyl, m. 162°; <sup>317</sup> 165°. <sup>573</sup>                              |
| Sorbic,                   | benzyl, m. 137°; <sup>359</sup> 184°. <sup>427</sup>                              |
| Phenylpropionic,          | benzyl, m. 186°. <sup>427</sup>   |
| Malonic,                  | <i>p</i> -bromobenzyl, m. 139°. <sup>293</sup>                                    |
| Methylmalonic,            | benzyl (monosalt), m. 146°. <sup>427</sup>  |
| Dimethylmalonic,          | benzyl (monosalt), m. 160°. <sup>427</sup>  |
| Diethylmalonic,           | benzyl (monosalt), m. 149°. <sup>427</sup>  |
| Phenylmalonic,            | benzyl, m. 145°. <sup>427</sup>   |
| Glutaric,                 | benzyl, m. 153°; <sup>427</sup> <i>p</i> -bromobenzyl, m. 149°. <sup>293</sup>    |



|                            |  |
|----------------------------|--|
| Adipic,                    | benzyl (monosalt), m. 164°; <sup>81</sup> benzyl, m. 163°. <sup>81</sup>   |
|                            | <i>p</i> -bromobenzyl, m. 167°. <sup>293</sup>   |
| Pimelic,                   | benzyl, m. 163°; <sup>427</sup> <i>p</i> -bromobenzyl, m. 149°. <sup>293</sup>   |
| Suberic,                   | benzyl, m. 151°. <sup>427</sup>  |
| Sebacic,                   | benzyl, m. 155°. <sup>81</sup>   |
| Acetylenedicarboxylic,     | benzyl, m. 160°. <sup>427</sup>  |
| Maleic,                    | benzyl, m. 163°; <sup>317</sup> 186°. <sup>124</sup>   |
| Fumaric,                   | benzyl, m. 178°. <sup>317</sup>  |
| Anisic,                    | benzyl, m. 177°. <sup>317</sup>  |
| <i>o</i> -Nitrobenzoic,    | benzyl, m. 159°. <sup>317</sup>  |
| <i>m</i> -Nitrobenzoic,    | benzyl, m. 163°. <sup>317</sup>  |
| <i>p</i> -Nitrobenzoic,    | benzyl, m. 182°. <sup>317</sup>  |
| 3,4-Dinitrobenzoic,        | benzyl, m. 152°. <sup>427</sup>  |
| 3,5-Dinitrobenzoic,        | benzyl, m. 178°. <sup>427</sup>  |
| Vanillic,                  | benzyl, m. 166°. <sup>427</sup>  |
| Piperonylic,               | benzyl, m. 173°. <sup>427</sup>  |
| Picric,                    | benzyl, m. 187°; <sup>427</sup> 188°. <sup>573</sup>   |
| Monomethylphthalate,       | benzyl, m. 134°. <sup>427</sup>  |
| Monobutylphthalate,        | benzyl, m. 138°. <sup>427</sup>  |
| $\alpha$ -Naphthoic,       | benzyl, m. 148°. <sup>427</sup>  |
| Nicotinic,                 | benzyl, m. 157°. <sup>427</sup>  |
| Picolinic,                 | benzyl, m. 186°. <sup>427</sup>  |
| Benzenesulfinic,           | benzyl, m. 155°. <sup>781</sup>  |
| <i>o</i> -Toluenesulfinic, | benzyl, m. 179°. <sup>781</sup>  |
| <i>p</i> -Toluenesulfinic, | benzyl, m. 168°. <sup>781</sup>  |
| Methanesulfonic,           | benzyl, m. 149°. <sup>781</sup>  |
| Ethanesulfonic,            | benzyl, m. 113°. <sup>427</sup>  |
| Cyclohexanesulfonic,       | benzyl, m. 143°. <sup>1293</sup>   |
| Ethylenesulfonic,          | benzyl, m. 146°. <sup>1384</sup>   |
| Benzenesulfonic,           | benzyl, m. 144°; <sup>317</sup> <i>p</i> -chlorobenzyl, m. 184°. <sup>294</sup>  |
| <i>o</i> -Phenolsulfonic,  | benzyl, m. 118.9°; <sup>938</sup> <i>p</i> -chlorobenzyl, m. 181°. <sup>124</sup>  |
| <i>p</i> -Phenolsulfonic,  | benzyl, m. 171.3°. <sup>938</sup>  |
| <i>m</i> -Sulfobenzoic,    | benzyl, m. 133°. <sup>427</sup>  |
| <i>o</i> -Toluenesulfonic, | benzyl, m. 180.8°. <sup>938</sup>  |
| <i>p</i> -Toluenesulfonic, | benzyl, m. 178°; <sup>317</sup> 179°; <sup>781</sup> 181°; <sup>573</sup> 182.2°; <sup>938</sup> <i>p</i> -chlorobenzyl, m. 193°. <sup>124</sup> |
| $\alpha$ -Toluenesulfonic, | benzyl, m. 127°. <sup>116</sup>  |

|   |                        |
|---|------------------------|
| $\alpha$ -Naphthalenesulfonic, benzyl, m.         | 138.5°. <sup>937</sup> |
| $\beta$ -Naphthalenesulfonic, benzyl, m.          | 192.3°. <sup>937</sup> |
| $\beta$ -Naphthol- $\alpha$ -sulfonic, benzyl, m. | 132°. <sup>427</sup>   |
| $\alpha$ -Naphthionic, benzyl, m.                 | 104°. <sup>549</sup>   |
| Quinoline-8-sulfonic, benzyl, m.                  | 250°. <sup>427</sup>   |

#### ALKYL S-BENZYLISOTHIURONIUM SULFATES <sup>47</sup>



R

|                   |            |
|-------------------|------------|
| Propyl,           | m. 112.5°. |
| <i>i</i> -Propyl, | m. 143°.   |
| Butyl,            | m. 101°.   |
| <i>i</i> -Butyl,  | m. 137°.   |
| <i>s</i> -Butyl,  | m. 119°.   |
| Amyl,             | m. 86°.    |
| Hexyl,            | m. 86°.    |
| Heptyl,           | m. 79°.    |
| Octyl,            | m. 47°.    |
| Decyl,            | m. 75°.    |
| Lauryl,           | m. 76°.    |
| Myristyl,         | m. 88°.    |
| Cyclohexyl,       | m. 164°.   |
| Bornyl,           | m. 175°.   |

#### S-BENZYLISOTHIURONIUM SALTS OF ALDEHYDE AND KETONE BISULFITES

Aldehyde <sup>1335</sup>

|                    |          |
|--------------------|----------|
| Propionic,         | m. 112°. |
| Butyric,           | m. 115°. |
| <i>i</i> -Valeric, | m. 117°. |
| Heptylic,          | m. 119°. |
| Caprylic,          | m. 118°. |
| Benzal,            | m. 113°. |
| Vanillin,          | m. 75°.  |

Ketone <sup>1038</sup>

|                |          |
|----------------|----------|
| Acetone,       | m. 140°. |
| Methylethyl,   | m. 68°.  |
| Acetophenone,  | m. 190°. |
| Cyclohexanone, | m. 120°. |

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## CHAPTER 2

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# Thiosemicarbazide

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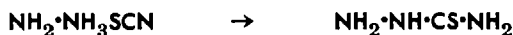
### Introduction

Thiosemicarbazide is usually written  $\text{NH}_2\cdot\text{CS}\cdot\text{NH}\cdot\text{NH}_2$  but, like thiourea, it may react as if it contained the  $-\text{SH}$  group. Two tautomeric formulae may be written:  $\text{HN}:\text{C}(\text{SH})\cdot\text{NH}\cdot\text{NH}_2$  and  $\text{H}_2\text{N}\cdot\text{C}(\text{SH})\cdot\text{N}\cdot\text{NH}_2$ . We have the progression:  $\text{NH}_2\cdot\text{CS}\cdot\text{NH}_2$ ,  $\text{NH}_2\cdot\text{CS}\cdot\text{NH}\cdot\text{NH}_2$ , and  $\text{H}_2\text{N}\cdot\text{NH}\cdot\text{CS}\cdot\text{NH}\cdot\text{NH}_2$ . That is, thiosemicarbazide is a hybrid, derived from ammonia and hydrazine. Numbering starts with the  $-\text{NH}_2$  of the hydrazine end:  $\text{NH}_2\cdot\text{CS}\cdot\text{NH}\cdot\text{NHPh}$  is 1-phenylthiosemicarbazide.<sup>369</sup>

### Thiosemicarbazide

#### PREPARATION

Thiosemicarbazide and its derivatives can be prepared by reactions that are closely analogous to those that produce thiourea and its derivatives. Hydrazine thiocyanate isomerizes to thiosemicarbazide under the same conditions as those under which ammonium thiocyanate goes to thiourea:



This may be regarded as the addition of hydrazine across a double bond to thiocyanic acid:



The transformation takes place under somewhat the same conditions but it has not been thoroughly studied.<sup>370</sup>

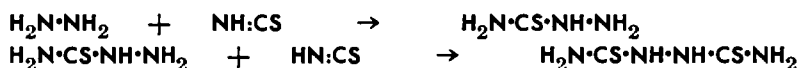
Evaporation of a hydrazine thiocyanate solution *in vacuo* below 100° gives a good yield of pure thiosemicarbazide.<sup>400</sup> A dispersion of hydrazine thiocyanate in an organic liquid may be heated.<sup>86</sup> Thiosemicarbazide is obtained by refluxing a solution of ammonium thiocyanate and hydrazine.<sup>16, 423, 448</sup> The hydrazine thiocyanate may be formed by double decomposition from a salt of hydrazine and a salt of thiocyanic acid.<sup>25, 156, 160a, 266, 361b, 463a, 463b, 506</sup> Potassium thiocyanate containing radioactive sulfur has been used to prepare radioactive thiosemicarbazide.<sup>91</sup>

Hydrazine sulfate added to a mixture of thiourea and calcium<sup>474a</sup> or sodium carbonate,<sup>474b</sup> gives thiosemicarbazide. Thiourea nitrate, reduced electrolytically in sulfuric acid solution, yields thiosemicarbazide sulfate.<sup>278</sup>

The addition of two molecules of thiocyanic acid to one of hydrazine gives the double compound,<sup>101</sup> the thiocarbazine: <sup>156, 160a</sup>

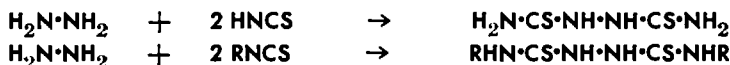


This reaction may be considered as taking place in two stages:



Isomeric forms,  $\text{H}_2\text{N}\cdot\text{CS}\cdot\text{NH}\cdot\text{NH}\cdot\text{C}(\text{:NH})\text{SH}$ , m. 223° and  $\text{HN:C}(\text{SH})\cdot\text{NH}\cdot\text{NH}\cdot\text{C}(\text{:NH})\text{SH}$ , m. 203°, have been isolated. Either can be changed into the other.<sup>246a</sup>

Both amino groups of hydrazine may be made to react by using two molecules of ammonium thiocyanate, or two of an alkyl isothiocyanate: <sup>156, 162b</sup>



An unsymmetrical dialkyl derivative results when a mustard oil reacts with a 4-alkylthiosemicarbazide: <sup>61</sup>



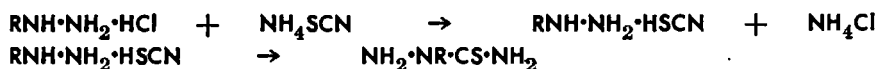
Thiosemicarbazide and cyanic acid give the monothio compound,  $\text{H}_2\text{N}\cdot\text{CS}\cdot\text{NH}\cdot\text{NH}\cdot\text{CO}\cdot\text{NH}_2$ .<sup>160b, 461</sup> A derivative of this can be made from semicarbazide and an isothiocyanate: <sup>197</sup>



The methyl derivative melts at 212° and the allyl at 202°. <sup>197</sup>



Similarly the thiocyanates of substituted hydrazines rearrange into 1- or 2-substituted derivatives of thiosemicarbazide: 14, 34, 82, 83a, 144, 175, 177b, 194a, 199, 303, 342, 357, 426, 428, 455b, 458, 509a

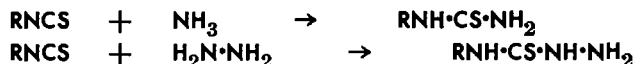


OR



1-Substituted thiosemicarbazide has been made by diazotizing sulfanilamide and coupling with ammonium thiocyanate.<sup>75</sup>

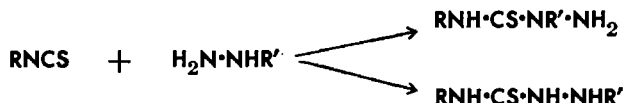
An isothiocyanate takes up ammonia or hydrazine to form a substituted thiourea or thiosemicarbazide: 52, 140, 152, 161a, 203, 293, 385, 498



Hydrazine hydrate is added slowly, with cooling, to an equivalent amount of phenyl mustard oil in alcoholic solution. The phenylthiosemicarbazide separates out as crystals which are practically pure.<sup>385</sup>

Two molecules of phenylisothiocyanate may be added to carbohydrazide giving carbohydrazidodicarbonthiophenylamide,  $\text{OC}(\text{NH}\cdot\text{NH}\cdot\text{CS}\cdot\text{NHPh})_2$  m.  $209^\circ$ .<sup>198</sup>

The addition of a mustard oil to a substituted hydrazine may give two isomeric compounds: 18, 46, 57a, 59, 62, 63, 65, 97, 126, 143a, 171b, 175, 200, 225, 230a, 286, 296, 307, 311, 442b, 442c, 466, 471, 483a, 483b, 513

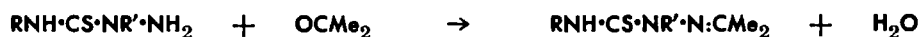


The relative amounts of the two depend on the reactants, the solvents, and the temperature.<sup>57a, 63</sup> The isomer having the substituent at the end is the more stable. In some cases the less stable isomerizes: 59, 62, 171b



1-Methyl-2,4-diphenylthiosemicarbazide rearranges to the 2-methyl-1,4-diphenyl-compound when heated to  $150^\circ$ .<sup>60</sup> There is uncertainty as to the structure of some compounds that have been reported. Sometimes both isomers can be isolated from the

reaction product.<sup>57a, 63</sup> Of the two isomers, only the one having the  $\text{-NH}_2$  group at the end reacts with an aldehyde or ketone to form a thiosemicarbazone: <sup>62</sup>

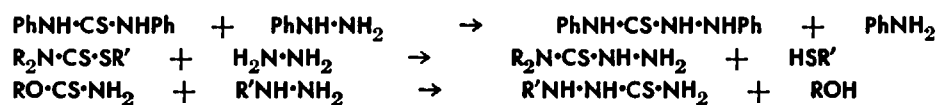


Yohimbic acid hydrazide and phenyl isothiocyanate treated with alcoholic HCl give the phenylthiocarbamyl hydrazide of yohimbic acid as the hydrochloride.<sup>240b</sup> The bis(4-phenylthiosemicarbazide) of N-acetyl- $\beta$ -amino adipic acid has been prepared from the corresponding acid hydrazide.<sup>269</sup> An isonitrile and sulfur may serve as the isothiocyanate.<sup>297</sup> A 1,4-dibenzoyl thiosemicarbazide having radioactive carbon in the carbonyl group of the 1-position has been prepared.<sup>344</sup>

The addition of an isothiocyanate to a disubstituted hydrazine gives a trisubstituted thiosemicarbazide: <sup>60, 224b, 226, 298a, 298b</sup>



Hydrazine, or a substituted hydrazine, may displace an amine from a substituted thiourea,<sup>223, 391</sup> a mercaptan from a dithiocarbamate,<sup>134, 249b</sup> or an alkoxy group from a xanthogenamide: <sup>309, 399</sup>



## REACTIONS

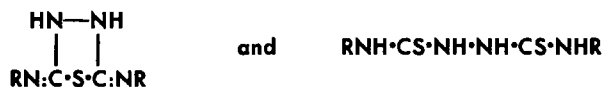
Hydrazine may be eliminated from two molecules of thiosemicarbazide: <sup>160a</sup>



Oxidation by alkaline hydrogen peroxide converts thiosemicarbazide to semicarbazide.<sup>267</sup> With sodium hypobromite the oxidation is more complicated, one hydrazo group is eliminated and one  $\text{-SH}$  is oxidised to the sulfonic acid. The product is hydrazodicarboxthioamide sulfonic acid,  $\text{HSC}(\text{:NH})\text{NH}\cdot\text{NHC}(\text{:NH})\text{SO}_3\text{H}\cdot\text{H}_2\text{O}$ , m.  $247^\circ$ .<sup>295</sup> The hydrazino group of thiosemicarbazide is oxidised to nitrogen by  $\text{NaOCl}$ .<sup>434</sup> In acid solution the titration of one molecule of thiosemicarbazide requires ten atoms of iodine; in alkaline solution it requires twelve.<sup>176</sup>

When a thiosemicarbazide, substituted in the 4-position, is

treated with sulfur monochloride, several reactions take place, which result in the formation of a sulfur diazole and the double compound: <sup>389</sup>



By heating it with an alkyl halide, thiosemicarbazide is alkylated on the sulfur atom just as thiourea would be: <sup>159</sup>



The same is true of its alkyl, <sup>83b</sup>, <sup>194a</sup>, <sup>303</sup> aryl, <sup>201</sup> and acyl, <sup>194b</sup>, <sup>230b</sup>, <sup>230c</sup> derivatives. Phenylldiazonium chloride acts like an alkyl halide with 1,4-diphenylthiosemicarbazide, giving the S-phenyl derivative  $\text{PhNH}\cdot\text{NH}\cdot\text{C}(\text{SPh})\cdot\text{NPh}$ , <sup>68</sup> but 2,4-dinitro-chlorobenzene attacks the 1-nitrogen atom of thiosemicarbazide, forming 2,4-(NO<sub>2</sub>)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>NH·NH·CS·NH<sub>2</sub>. <sup>188</sup>

Refluxing thiosemicarbazide with formic acid gives the 1-formyl derivative, which is changed by acetyl chloride into 2-amino-1,3,4-thiadiazole. <sup>158</sup>, <sup>190</sup> The corresponding acetyl derivative behaves similarly. <sup>158</sup> A 1-acyl derivative of a 4-substituted thiosemicarbazide is converted by heat alone to a 5-mercapto-1,2,4-triazole but is dehydrated by acetyl chloride to a derivative of 2-substituted-amino-5-alkyl-1,3,4-thiadiazole. <sup>154b</sup>, <sup>306</sup> The 1-formyl derivative of 4-phenylthiosemicarbazide can be converted into 2-phenyl-amino-1,3,4-thiadiazole; <sup>386</sup> so with the 4-allyl. <sup>386</sup>

1,4-Diphenylthiosemicarbazide and acetyl chloride form what appears to be an addition compound, m. 233°, which acts like a salt of a moderately strong base. It loses hydrochloric acid, becoming the acetyl derivative,  $\text{PhNH}\cdot\text{CS}\cdot\text{NH}\cdot\text{NAcPh}$ . This is also formed by the addition of phenyl isothiocyanate to acetylphenylhydrazine. <sup>304</sup> The benzoylation of thiosemicarbazide has been studied; <sup>442a</sup>, <sup>452</sup> its thiobenzoylation has been accomplished. <sup>251</sup>, <sup>283</sup> Acyl chlorides give 1-acyl thiosemicarbazides. <sup>358</sup> The 2-furoyl-, <sup>332</sup> 2-thenyl-, <sup>505</sup> and 4-pyridyl- <sup>505</sup> derivatives have been prepared similarly. Acylation has been effected by acetanhydride <sup>262</sup> and quinolinic anhydride. <sup>212</sup> Substituted thiosemicarbazides react with malonyl dichloride in the presence of pyridine, giving substituted 1,2,4-heptatriazine-5,7-diones. <sup>301</sup>

Ethyl chloroformate and 4-phenylthiosemicarbazide give the

ester,  $\text{PhNH}\cdot\text{CS}\cdot\text{NH}\cdot\text{NH}\cdot\text{COOEt}$ . Boiling this with alkali converts it into 3-hydroxy-4-phenyl-5-thiatriazole. Carbethoxythiosemicarbazide,  $\text{H}_2\text{N}\cdot\text{CS}\cdot\text{NH}\cdot\text{NH}\cdot\text{COOEt}$ , is formed similarly.<sup>168</sup>

2,4-Diphenylthiosemicarbazide condenses with phosgene to give 1-phenylthiobiazolone-5-anil.<sup>60</sup> Conditions have been described for a reaction between thiosemicarbazide and chloroacetone to yield 3-amino-2-imino-4-methyl-4-thiazoline hydrochloride.<sup>42</sup> Thiosemicarbazide, or a 4-substitution product, reacts with  $\omega$ -bromoacetophenone to give a thiadiazine and a thiazole, the relative proportions of which depend on the groups present.<sup>50</sup> With  $\alpha$ -chloroacetoacetic ester the product is apparently bicyclic.<sup>51b</sup> Thiosemicarbazide and ethyl chloroacetate give 2-amino-5-hydroxy-1,3,4-thiadiazine, m.  $284^\circ$ . The 2-methyl homolog melts at  $282^\circ$ , the 2-ethyl at  $225^\circ$ , and the 2-*i*-butyl at  $210^\circ$ .<sup>51a</sup> A thiadiazine is formed from a thiosemicarbazide and  $\alpha$ -chloroacetamide.<sup>489</sup>

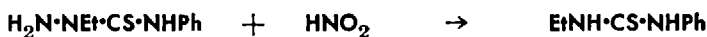
The cyclization of substituted thiosemicarbazides to diazole and triazole derivatives has been extensively investigated.<sup>187, 294, 333, 509b</sup> A series of heterocyclic compounds, the hydrazinothiazolinones, arising from the action of  $\alpha$ -haloacids on thiosemicarbazides or the thiosemicarbazones of aldehydes has been described.<sup>88</sup>  $\beta$ -Oxo-carboxylic acid esters and  $\beta$ -dioxo compounds give ring products with thiosemicarbazide and 2-substituted thiosemicarbazides.<sup>300</sup>

According to the conditions under which they are brought together, thiosemicarbazide and  $\text{PhCCl:NPh}$  yield 3,4,5-triphenyl-triazole hydrochloride or 1,4,5-triphenyl-3,5-endothiodihydro-1,2,4-triazole hydrochloride. The free base melts at  $315^\circ$ .<sup>67</sup>

1-Phenylthiosemicarbazide reacts with  $\alpha,\beta$ -dichlorodiethyl ether and thiazoles are the chief products.<sup>40</sup>

S-Methyl-2-methylthiosemicarbazide,  $\text{HN:C(SMe)NMeNH}_2$ , condenses with phenylpyruvic acid to 2-methyl-3-methylmercapto-5-keto-6-benzyl-2,5-dihydro-1,2,4-triazine, m.  $116.5^\circ$ .<sup>83b</sup>

Nitrous acid may convert a thiosemicarbazide into a thio-urea: <sup>65</sup>



This was used to prove that what Fischer supposed to be 1-ethyl-4-phenyl-thiosemicarbazide really is the 2-ethyl-4-phenyl-thiosemicarbazide. This conclusion was confirmed by the formation

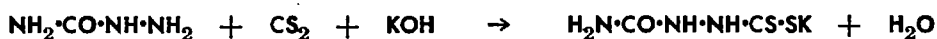
of a thiosemicarbazone.<sup>65, 143b</sup> Under certain conditions the product is a 5-amino-1,2,3,4-thiatriazole.<sup>160a, 407</sup> A 4-alkyl derivative gives the corresponding 5-alkylamino derivative, but a 4-aryl-thiosemicarbazone goes to 1-aryl-5-thiatetrazole.<sup>159, 161b</sup>

When allylthiosemicarbazide is treated with hydrochloric acid, hydroxymethylthiazole is produced; but if the 1-position is occupied by certain groups either a thiazole or a thiadiazole ring is formed.<sup>106</sup> Urea and a 4-alkylthiosemicarbazide give four different products, three of them being triazole derivatives.<sup>204</sup> Aromatic amines convert thiosemicarbazides into derivatives of 1,2,4-triazole.<sup>315d</sup>

Thiosemicarbazide reacts like an amine with carbon disulfide and an alkali: <sup>9b, 196b</sup>



S-Methylthiosemicarbazide gives the analogous compound,  $\text{H}_2\text{N}\cdot\text{C}(\text{SMe})\cdot\text{N}\cdot\text{NHCS}_2\text{K}$ .<sup>9b</sup> Semicarbazide reacts similarly with carbon disulfide and alkali:



The methyl ester, derived from this by methylation, forms a potassium salt,  $\text{H}_2\text{N}\cdot\text{CO}\cdot\text{NH}\cdot\text{N}:\text{C}(\text{SK})\text{SMe}$ , which reacts further with methyl iodide to form  $\text{H}_2\text{N}\cdot\text{CO}\cdot\text{NH}\cdot\text{N}:\text{C}(\text{SMe})_2$ .<sup>58</sup>

Thiosemicarbazide loses hydrogen sulfide when it is refluxed with alkali: <sup>407</sup>



Thiosemicarbazide and acetodinitrile form thiouramidoacetodinitrile, of which there are two isomeric forms. One of these melts at 106°; the other melts at 122°, resolidifies, and melts at 162°.<sup>318</sup>

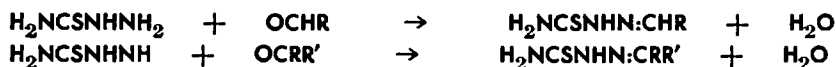
Thiosemicarbazide, hydrazodithiourea,  $\text{H}_2\text{N}\cdot\text{CS}\cdot\text{NH}\cdot\text{NH}\cdot\text{CS}\cdot\text{NH}_2$ , and its mono- and diphenyl derivatives, when refluxed with aniline or a toluidine, form derivatives of 1,2,4-triazole.<sup>315b</sup>

4-Phenylthiosemicarbazide and ethyl xanthate condense to 5-phenylimino-2-thio-tetrahydrothiadiazole.<sup>203</sup> Acetylurethane with thiosemicarbazide gives 1-carbamido-3-methyl-5-keto-1,2,4-triazole, m. 222°. With 4-allylthiosemicarbazide the product is the 1-allylcarbamido-analog, m. 201°.<sup>184</sup>

## Thiosemicarbazones

## PREPARATION

Thiosemicarbazide, like its oxygen analog, reacts with aldehydes and ketones, giving derivatives many of which are crystalline and suitable for purposes of identification:



They have been employed extensively: 65, 72, 127, 145, 146, 160c, 315a, 346, 356b, 373, 396, 406, 415, 467a, 467b, 467c, 484b, 496, 512. A number of these derivatives may be found in the table of properties at the end of the chapter. Hydrazine thiocyanate may be substituted for thiosemicarbazide in the preparation of the thiosemicarbazone.<sup>444a</sup>

An improved method for preparing thiosemicarbazones from ketones has been claimed.<sup>139</sup> An oxime of a pyruvic ester<sup>189</sup> and a ketazine<sup>331a</sup> react with thiosemicarbazide to form thiosemicarbazones. A thiosemicarbazone may be obtained by heating a semicarbazone with an aqueous solution of thiosemicarbazide hydrochloride.<sup>245</sup> Another method is the reaction of thiocyanic acid<sup>330</sup> or an isothiocyanate<sup>54</sup> on the hydrazone of an aldehyde:

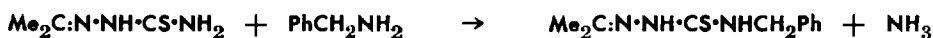


A thiosemicarbazone may be prepared from a dihydrazone and ammonium isothiocyanate.<sup>506</sup> Variations of the above methods and others have been described.<sup>19, 41, 43, 131i, 331b, 411, 446, 447</sup> Thiosemicarbazones have been prepared from a number of alkylcyclohexenones.<sup>315c</sup> 4-*o*-Tolylthiosemicarbazide reacts with aldehydes.<sup>167</sup>

## REACTIONS

The sodium derivative of acetone thiosemicarbazone gives the S-benzyl derivative with benzyl chloride. This with alkali liberates benzyl mercaptan; with hydrochloric acid it is hydrolyzed to S-benzylthiosemicarbazide.<sup>495</sup> Acetone thiosemicarbazone treated with sodium ethylate and then with alkyl halides gives S-alkyl derivatives. Acetone thiosemicarbazone is converted to

the 4-benzyl derivative by boiling in toluene solution with benzylamine: <sup>24</sup>



There are two thiosemicarbazones from camphor, one of which is unstable and goes into camphanethiotriazine, m. 207°. <sup>148</sup>

Oxidation of benzalthiosemicarbazone by ferric chloride or hydrogen peroxide gives 5-phenyl-2-amino-1,3,4-thiadiazole. <sup>108</sup> Similarly 5-phenyl-3-methylthiol-1,2,4-triazole, m. 166°, is obtained from the S-methyl derivative. <sup>107</sup> The oxidation of other thiosemicarbazones and the products have been described. <sup>122, 235</sup> Thiosemicarbazones are desulfurized to the corresponding semicarbazones by alkaline hydrogen peroxide. <sup>267</sup>

When there is a substituent on the 1-nitrogen, condensation with an aldehyde or ketone gives a ring compound instead of a thiosemicarbazone. <sup>57b, 64</sup>

Compounds of the type  $\text{RR}'\text{CHNHNHCSNH}_2$ , where R is an aryl group and R' is H, Me, Et,  $\text{CO}_2\text{H}$ , or Ph, are prepared by reduction of the corresponding thiocarbazones. <sup>131a</sup> Thiosemicarbazones of many aldehydes have been converted to heterocyclic compounds by  $\alpha$ -haloacids notably chloroacetic acid. <sup>87, 459, 476, 484a, 484c</sup> Two molecules of a thiosemicarbazone react almost instantaneously with one molecule of benzoyl peroxide to give a disulfide. <sup>228</sup> Those of  $\alpha$ -oxo acids yield 1,2,4-triazine derivatives when treated with dilute alkali. <sup>179.5b</sup> Thiosemicarbazones of pyridine aldehydes form  $\text{N}^4$ ,S-diacetyl derivatives that undergo oxidative cyclization to 2-pyridyl-4-acetamido-1,3,4-thiadiazoles. <sup>222</sup>

### Complexes and Analytical Reactions

Thiosemicarbazide and its derivatives resemble thiourea in their ability to form complexes with salts of heavy metals such as nickel, <sup>249a, 252</sup> cobalt, <sup>252</sup> copper, <sup>210, 252, 277, 381, 382</sup> palladium, <sup>249a</sup> platinum, <sup>420</sup> and silver. <sup>219</sup> These complexes may serve for the detection and estimation of platinum, <sup>13, 28, 29, 275, 472</sup> ruthenium, <sup>27</sup> osmium, <sup>27</sup> and of selenites and selenates. <sup>317</sup>

The 1-quinolyl-4-allylthiosemicarbazide,  $\text{C}_3\text{H}_5\text{NH}\cdot\text{CS}\cdot\text{NH}\cdot\text{NH}\cdot\text{C}_9\text{H}_6\text{N}$ , is a sensitive precipitant for cadmium ions and it can be used for detecting cadmium ions in the presence of copper. <sup>420</sup> Several others give colored complexes with cobalt, copper, and mercury salts and may be used as micro-analytical reagents. <sup>31</sup> 1-

Allyl-4-*p*-nitrophenylthiosemicarbazide gives a red precipitate with mercuric ions at one part per million and a color with one tenth that concentration.<sup>421</sup> 1-Phenylthiosemicarbazide gives color tests with several metallic ions.<sup>341, 342, 343</sup> It may be used for the detection and determination of mercurous, mercuric,<sup>276a, 276b</sup> platinum,<sup>273, 340</sup> molybdenum,<sup>274</sup> and ruthenium<sup>270, 503</sup> ions. A diphenylthiosemicarbazide has been recommended as a reagent for the detection of rhenium ions.<sup>180</sup>

Mercuric nitrite forms complex salts with thiosemicarbazide, and 1,4-diphenylthiosemicarbazide.<sup>394</sup> The relation of constitution to the formation of complexes has been studied.<sup>141</sup> A substituted thiosemicarbazide may be used to counteract the catalytic oxidation of cracked gasoline by traces of heavy metals.<sup>93</sup>

Thiosemicarbazide reduces a chromate and precipitates the chromium as the hydroxide.<sup>217</sup>

Thiosemicarbazide may be titrated by a hydrochloric acid solution of iodine containing either persulfate<sup>433</sup> or mercuric chloride.<sup>432</sup> Potentiometric methods of titration have been proposed.<sup>90a, 90b, 328</sup> A color reaction with sodium nitroprusside may be used for detection.<sup>33</sup>

Thiosemicarbazones also form complexes with heavy metals.<sup>21, 236, 238</sup> Comprehensive studies have been made of these as analytical reagents.<sup>45, 205, 238, 329, 378, 422</sup> The derivatives of certain  $\alpha$ -diketones form colored complexes with copper, nickel, palladium, and zinc.<sup>22, 23</sup> The silver complex of 4-acetamidobenzaldehyde thiosemicarbazone can be dried and weighed.<sup>220</sup> Biacetyl-oxime-thiosemicarbazone is a reagent for the detection of manganous ions.<sup>237</sup> The 4-allylthiosemicarbazone of *p*-hydroxybenzaldehyde is useful in testing for bismuth.<sup>422</sup> The volatility of polonium thiosemicarbazone compounds has been investigated.<sup>305</sup>

Thiosemicarbazones may be separated by paper chromatography.<sup>150</sup> Reactions for their detection and determination have been reviewed.<sup>211, 284, 416</sup> They may be titrated with ferric chloride<sup>502b</sup> or iodine.<sup>326</sup> An argentometric method has been proposed.<sup>322</sup> They may be determined colorimetrically by Grote's reagent<sup>328</sup> or by (2-diethylaminoethyl)-1-naphthylamine oxalate.<sup>477</sup> Thiosemicarbazones give colors with dimethylglyoxime and hydrochloric acid under certain conditions.<sup>356a</sup> The thiosemicarbazone of *p*-acetaminobenzaldehyde has received special attention because of its therapeutic value. Methods for estimating



it have been compared.<sup>285</sup> It may be titrated with silver nitrate or bromide-bromate.<sup>409</sup> Its silver complex may be weighed.<sup>866</sup> A polarographic method has been described.<sup>123</sup> A gravimetric method using 2,4-dinitrophenylhydrazine has been proposed.<sup>837</sup> Methods for estimating thiosemicarbazones in the blood<sup>436a</sup> and urine<sup>502a</sup> have been given. Preliminary experiments to identify and estimate thiosemicarbazones of aromatic aldehydes in pharmaceutical preparations by studies of ultraviolet absorption indicate this technique can be used in certain cases.<sup>243</sup> The 4-allylthiosemicarbazones of certain aromatic aldehydes give color tests with heavy metal ions.<sup>422</sup>

### Other Applications

The therapeutic uses of thiosemicarbazide derivatives have been explored<sup>4, 131g, 136, 138d, 287, 469, 510</sup> particularly with reference to tuberculosis. A number of substituted thiosemicarbazides,<sup>3, 8b, 137 alkyl,<sup>467e</sup> aryl,<sup>8a, 75, 80, 82, 135, 171b, 182, 231, 479</sup> and N-acylated aryl<sup>71, 78a, 491</sup> have been studied. Sulfonamide salts of some of these are said to be useful.<sup>32a, 131b</sup></sup>

The chief interest has been in the thiosemicarbazones,<sup>1, 32c, 38, 115, 214, 221, 232, 247, 290, 313, 323, 338, 355b, 439, 492</sup> on which there are three reviews.<sup>81, 325, 430</sup> Such compounds have been prepared from glucose,<sup>256</sup> unsaturated aldehydes,<sup>281</sup> benzaldehyde,<sup>114, 456</sup> substituted benzaldehydes,<sup>30, 32b, 37b, 55, 70, 99, 102, 131c, 131e, 132, 133b, 133c, 133d, 133g, 138a, 138b, 170, 174, 191, 211, 218, 242, 244, 260, 291, 299a, 299b, 349a, 349b, 349c, 351, 354, 355a, 359, 360, 362, 368, 372, 390, 392, 395, 413b, 425, 435, 453, 455a, 468, 473, 489.5, 493, 497, 506</sup> other aromatic aldehydes,<sup>77a, 77b, 103, 131d, 131f, 131h, 149b, 240a, 272, 413a, 467d</sup> pyridine aldehydes,<sup>117, 177a, 179.5a, 213a, 229, 445</sup> other heterocyclic aldehydes,<sup>6, 32d, 94, 104, 118, 121, 133e, 133f, 133h, 149a, 178, 213b, 279, 280, 282, 403, 404, 424, 460, 490</sup> and also from ketones.<sup>35, 74, 131g, 138c, 207, 255, 353, 376, 414a, 414b</sup>

Certain thiosemicarbazones can be converted to azomethine compounds which are said to have special anti-tubercular properties.<sup>138e</sup> The condensation of  $p\text{-OHCC}_6\text{H}_4\text{CH:NNHCSNH}_2$  with  $\text{N}_2\text{H}_4$  and its derivatives yield hydrazones and azimes which are claimed to be useful in the treatment of tuberculosis, leprosy, arthritis, and as a fungicide.<sup>131h</sup> Several metal complexes of thiosemicarbazones have been investigated.<sup>36, 417</sup>

One compound, the thiosemicarbazone of  $p$ -acetaminobenzaldehyde has received special attention and has been named Tibione.

There are a number of favorable reports on its efficiency.<sup>96, 113a, 113c, 206, 208, 254, 257, 261, 308, 310, 313, 314, 339, 345, 352, 389, 481, 487, 508</sup>

Several of its derivatives have been studied.<sup>37a, 112, 133a, 259, 443</sup>

Various physiological effects of thiosemicarbazide<sup>105, 248, 264, 265, 348, 350, 365, 371, 388, 401, 429, 431, 494a, 499</sup> and of its derivatives<sup>7, 39, 76, 84, 95, 113b, 248, 375, 393, 402a, 402b, 419, 431</sup> have been reported.

The side effects from the administration of thiosemicarbazones have been investigated.<sup>129, 209, 227, 234, 271, 289, 380, 398, 408, 470, 475, 485, 511</sup> They are not recommended for children under six years old.<sup>494b</sup>

Thiosemicarbazide shows antihypertensive activity.<sup>418</sup> A number of its derivatives inhibit sarcoma in mice.<sup>153</sup> Diphenylthio-carbazone has been used in an experimental study of diabetes in the rabbit.<sup>363</sup> Several thiosemicarbazide derivatives have been found to have bactericidal<sup>98, 319</sup> and antiviral<sup>73b</sup> action. A number of thiosemicarbazones have been tested as bactericides.<sup>85, 113d, 172a, 172b, 172c, 183, 250, 261, 310, 349d, 374, 465</sup> Several have antiviral activity.<sup>47, 78, 192, 215, 216, 302, 327, 464, 482</sup>

Thiosemicarbazide<sup>169, 179</sup> derivatives of 2-phenyl-thiosemicarbazide,<sup>316</sup> and  $\beta$ -naphthaldehyde thiosemicarbazone<sup>383</sup> have fungicidal properties.

In a dilution of 1 to 10,000 thiosemicarbazide killed 98% of certain larvae.<sup>79</sup> Certain aromatic thiosemicarbazides are toxic to insects.<sup>53b, 449</sup> 4-Phenylthiosemicarbazones of some aldehydes have been claimed as insecticides.<sup>53a</sup> The insecticide effect of the thiosemicarbazone group has been evaluated.<sup>151</sup>

Thiosemicarbazide<sup>110, 488</sup> and its phenyl-,<sup>258</sup> and *o*-tolyl-<sup>111</sup> derivatives have been tested as rodenticides.

Thiosemicarbazide breaks the dormancy of potato tubers.<sup>324</sup> Certain thiosemicarbazones favor the early growth of buds.<sup>384</sup> The 2,4-dichlorobenzaldehyde thiosemicarbazone is a growth retarder.<sup>89</sup>

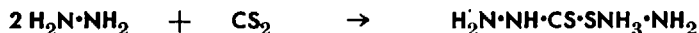
When thiosemicarbazide is heated with dicarboxylic acids, their anhydrides, or esters, it is said to form valuable polymers, some of which are fiber-forming.<sup>334, 377</sup> Thiosemicarbazide and some of its derivatives have been found of value in photography.<sup>168, 241, 263, 335</sup> Some of them counteract the deleterious effect of traces of heavy metals in certain materials.<sup>5, 92</sup> Diallyl-thiosemicarbazide in small concentrations promotes the oxidation of chlorophyll solutions, while in large concentrations it is a

stabilizer.<sup>486</sup> Several thiosemicarbazides are claimed as catalysts in the preparation of trans  $\alpha,\beta$ -unsaturated carboxylic acids.<sup>239</sup> Thiosemicarbazide is said to inhibit the dendritic crystallization of lead.<sup>379</sup> In making a solution of 17 parts polyacrylonitrile and 83 parts dimethylformamide, an increase in viscosity was prevented by adding 1% of thiosemicarbazide.<sup>336</sup> It has been used in the synthesis of a dye;<sup>437, 441</sup> some of its derivatives have been added to copper plating baths.<sup>454</sup>

### Thiocarbohydrazide

#### PREPARATION

Instead of just one, both of the amino groups of thiourea may be substituted by hydrazine residues. Carbon disulfide reacts with hydrazine hydrate as with ammonia, forming the hydrazine salt of dithiocarbazinic acid:



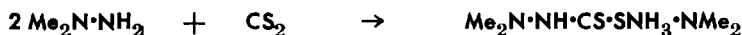
As is the case with the ammonium salt, this loses hydrogen sulfide: 15, 101, 440



Lead oxide is stirred into the aqueous solution of the salt which is heated on the water bath. The thiocarbohydrazide, m. 169°, crystallizes out of the filtrate.<sup>440</sup>

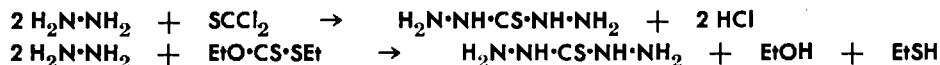
Thiocarbohydrazide may be prepared by refluxing hydrazinium dithiocarbozinate in a water solution of hydrazine.<sup>17, 361a</sup> There is a review of six papers on the compound which contains additional references.<sup>410</sup>

Dimethylhydrazine combines with carbon disulfide:



When a solution of this salt is acidified, the free acid  $\text{Me}_2\text{N}\cdot\text{NH}\cdot\text{CS}\cdot\text{SH}$ , m. 112°, is precipitated.<sup>397</sup> From 2-methyl semicarbazide and carbon disulfide the salt,  $\text{MeHN}\cdot\text{NH}\cdot\text{CS}\cdot\text{SNH}_3\cdot\text{NHMe}$  can be prepared and from it, 1,5-dimethylthiocarbohydrazide,  $\text{MeNH}\cdot\text{NH}\cdot\text{CS}\cdot\text{NH}\cdot\text{NHMe}$ .<sup>320</sup>  $\text{ArNH}\cdot\text{NH}\cdot\text{CS}\cdot\text{SNH}_3\text{NHAr}$  gives  $(\text{ArNH}\cdot\text{NH})_2\text{CS}$ .<sup>171a</sup>

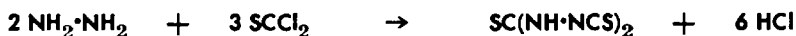
Thiocarbohydrazide results also when hydrazine reacts with thiophosgene or with ethyl xanthate: 198



Alkyl- and aryl-hydrazines react similarly: <sup>31</sup>



In hydrochloric acid solution the reaction goes differently and the free amino groups are attacked:



The product is 5-dithiocarbimidothiurea. From phenylhydrazine the diphenyl derivative is obtained. This undergoes a sort of disproportionation:



The isothiocyano groups can be made to migrate to the ring:



Thiophosgene changes this to 1,2,4- $\text{C}_6\text{H}_3(\text{NCS})_3$ . Different products,  $\text{SC}(\text{NH}\cdot\text{NH}\cdot\text{CONH}_2)_2$  m.  $215^\circ$  and  $\text{SC}[\text{N}(\text{NCS})\cdot\text{CO}\cdot\text{NH}_2]_2$ , are likewise obtained from semicarbazide and thiophosgene.<sup>31</sup>

### REACTIONS

Dimethylenethiocarbohydrazide,  $\text{SC}(\text{NH}\cdot\text{N}:\text{CH}_2)_2$ , m.  $205^\circ$ , is from thiocarbohydrazide and formaldehyde. When thiocarbohydrazide and potassium xanthate are heated to  $100^\circ$  in ethanol solution, dithio-*p*-urazine,  $\text{SC}(\text{NH}\cdot\text{NH})_2\text{CS}$ , m.  $203^\circ$ , is formed. Carbonylhydrazide, treated similarly, gives the monothio-,  $\text{SC}(\text{NH}\cdot\text{NH})_2\text{CO}$ , m.  $238^\circ$ . The dithio-*p*-urazine is formed when hydrazine hydrate, two molecules of carbon disulfide, and water are heated to  $150^\circ$ .<sup>198</sup>

Thiocarbohydrazide and ethyl chloroacetate are condensed by sodium ethylate to 3-amino-2,4-diketothiazolidine-2-hydrazone, m.  $120^\circ$ . The 5-alkyl derivatives are obtained from the esters of the higher halogen-acids.<sup>438b</sup>

Thiocarbohydrazide has various applications in analytical chemistry. It reacts with many anions and cations to produce colors and precipitates. It can be used to separate molybdenum from tungsten and uranium, to analyze molybdenum, to carry out reactions under the microscope, and to distinguish aldehydes and ketones.<sup>69, 124</sup> Thiocarbohydrazide can be employed in the determination of organic functionals as it precipitates many aldehydes and ketones completely.<sup>125</sup>

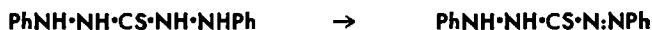
*s*-Diphenylthiocarbohydrazide,  $\text{PhNH}\cdot\text{NH}\cdot\text{CS}\cdot\text{NH}\cdot\text{NHPh}$ , is of

special interest since it is so easily oxidised to dithizone, which has come to be an important analytical reagent and will be considered in the next chapter.

Phenylhydrazine combines with carbon disulfide to the salt  $\text{PhNH}\cdot\text{NH}\cdot\text{CS}\cdot\text{SNH}_3\text{NHPh}$  from a solution of which the unstable acid,  $\text{PhNH}\cdot\text{NH}\cdot\text{CS}\cdot\text{SH}$ , can be precipitated. The salt loses hydrogen sulfide on heating:



This can be purified by recrystallization from alcohol but long heating must be avoided. Heated alone or in alcohol solution it is oxidised. In alkaline solution the oxidation is rapid and complete: <sup>143a</sup>



Other *s*-diarylthiocarbohydrazides may be prepared either by treatment of the corresponding aryl hydrazine with carbon disulfide; <sup>195, 462</sup> or by coupling 2 molecules of diazonium compound with methyl nitrite and reducing with ethanolic ammonium sulfide. <sup>462</sup>

Diphenylthiocarbohydrazide has been used for the colorimetric determination of magnesium.<sup>2, 210</sup> The same reagent has been employed for the estimation of bismuth <sup>119, 120, 367</sup> and lead.<sup>288</sup> It gives colors with heavy metal ions: orange with bismuth and lead; yellow, changing to green, with cobalt; dark green with copper; yellow with nickel and mercury; <sup>507</sup> and red, changing to brown, with silver.<sup>367</sup>

1,5-Diphenylthiocarbohydrazide exhibits *in vitro* antitubercular activity.<sup>480</sup>

Diphenylthiocarbohydrazide is converted into a cyclic compound by treatment with thionyl chloride.<sup>162a</sup> It condenses with phosgene, forming phenylazophenylthiobiazole.<sup>157</sup>

### Hydrazodicarboxthioamide

The formation of  $\text{H}_2\text{N}\cdot\text{CS}\cdot\text{NH}\cdot\text{NH}\cdot\text{CS}\cdot\text{NH}_2$  has been mentioned previously along with the preparation of thiosemicarbazide because several reactions give the one or the other according to the proportions of the reactants. The same may be said about the derivatives.

To prepare hydrazodicarboxthioamide: 50 g hydrazine sulfate

is added to a warm solution of 85 g potassium thiocyanate (2.27 moles) and the solution is boiled 2 hours. The compound is filtered off the next day and recrystallized from hot water; yield 50%. Thiosemicarbazide can be recovered from the mother liquor.<sup>156</sup> Heated with hydrochloric acid, it gives a mixture of dithiourazole and imidothiourazole.<sup>154a, 196a</sup>

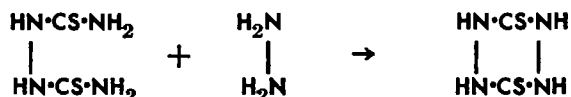
Di(thiocarbamyl)hydrazine is said to inhibit the growth of certain fungi.<sup>450</sup>

Oxidation of hydrazodithiurea,  $\text{H}_2\text{N}\cdot\text{CS}\cdot\text{NH}\cdot\text{NH}\cdot\text{CS}\cdot\text{NH}_2$  (A), by hydrogen peroxide gives 3,5-diamino-4,1,2-thiadiazole (B), whereas treatment of A with hydrazine results in 1-mercapto-4-amino,2,3,5,6-tetrazine (C).<sup>165</sup>



The di-S-methyl derivative of hydrazodicarboxthioamide,  $\text{HN}\cdot\text{C}(\text{SMe})\text{NH}\cdot\text{NH}\cdot\text{C}(\text{SMe})\cdot\text{NH}$ , is oxidised by potassium ferricyanide to the corresponding azo compound.<sup>11, 12</sup> Oxidation by bichromate gives methylsulfonyldithiourazole dimethyl ether.<sup>11</sup>

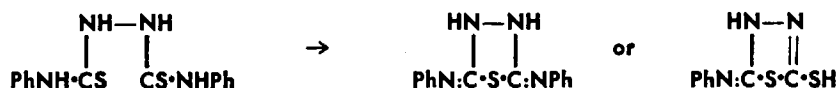
With hydrazine sulfate, a compound is formed which has been given a simple cyclic structure:



The properties indicate that it is polymeric.<sup>387</sup> With hydrazine hydrate the products are N(4) amino-imino-thiourazole and 4,4-diamino-hydrazothiourazole.<sup>9a</sup> With  $\alpha$ -bromo-butyric acid, 2,4-diketo-5-methyltetrahydrothiazole-2-ketazine is the product.<sup>438c</sup>

The condensation of  $\text{H}_2\text{NCSNHNHCONH}_2$  with  $\alpha$ -halogenated keto compounds gives 1-(2-thiazolyl)semicarbazides.<sup>44</sup>

When heated, the diphenyl derivative,  $\text{PhNH}\cdot\text{CS}\cdot\text{NH}\cdot\text{NH}\cdot\text{CS}\cdot\text{NHPh}$ , loses either hydrogen sulfide or aniline to give a ring compound:



The unsubstituted and other substituted hydrazodicarboxthio-

amides behave similarly. The presence of hydrochloric acid favors the elimination of aniline or ammonia, and a base aids the splitting out of hydrogen sulfide.<sup>10, 66, 154a, 155, 156, 163, 246b</sup> A triazole may also be formed.<sup>202</sup>

### Physical Properties of Thiosemicarbazide and Some of Its Derivatives

The resonance energy of thiosemicarbazide is 28 k.cal. per mole.<sup>444b</sup> Its heat of combustion is 4574 calories per gram and its heat of formation is  $5.7 \pm 0.5$  k.cal. per mole. The heat of solution for a 0.4% solution of thiosemicarbazide is 7.7 k.cal. per mole.<sup>444c</sup> In aqueous solution its dielectric constant does not change with dilution.<sup>109</sup> Infrared spectra of some of its 4-substituted derivatives have been investigated.<sup>292</sup>

Thiosemicarbazide melts at  $183^{\circ}$ ,<sup>25, 79, 156, 309</sup>  $182^{\circ}$  dec.,<sup>474a</sup>  $181.5^{\circ}$ ,<sup>474b</sup>  $180-3^{\circ}$ ,<sup>506</sup>  $180-2^{\circ}$ ,<sup>423</sup>  $180^{\circ}$ ,<sup>16</sup>  $179-81^{\circ}$ ,<sup>442c</sup>  $177-80^{\circ}$ .<sup>448</sup> Its hydrochloride melts at  $191^{\circ}$  with decomposition.<sup>442c</sup>

### MELTING POINTS OF SOME 1-DERIVATIVES



#### *Alkyl*

Methyl, m.  $248-50^{\circ}$ .<sup>483</sup>  
 $\alpha$ -Cyano-*i*-propyl, m.  $159^{\circ}$ .<sup>173</sup>  
 $\alpha$ -Carbamyl-*i*-propyl, m.  $201^{\circ}$ .<sup>173</sup>  
 $\alpha$ -Cyano-*s*-butyl, m.  $141^{\circ}$ ,<sup>173</sup>  $133-5^{\circ}$ .<sup>405</sup>  
*t*-Butyl, m.  $137.5^{\circ}$ .<sup>412</sup>  
 $\alpha$ -Carbamyl-*t*-butyl, m.  $190^{\circ}$ .<sup>173</sup>  
 Allyl, m.  $212-4^{\circ}$ ,<sup>483</sup>  $124.5^{\circ}$ .<sup>175</sup>  
 1-Cyanocyclohexyl, m.  $173^{\circ}$  dec.<sup>173</sup>  
 1-Carbamylcyclohexyl, m.  $205^{\circ}$  dec.<sup>173</sup>

#### *Aryl*

Phenyl, m.  $221-3^{\circ}$ ,<sup>483</sup>  $201^{\circ}$  dec.<sup>144</sup>  
*p*-Chlorophenyl, m.  $198^{\circ}$ .<sup>566</sup>  
*p*-Bromophenyl, m.  $110^{\circ}$ .<sup>203</sup>  
*o*-Nitrophenyl, m.  $200^{\circ}$  dec.<sup>199</sup>  
 2,4-Dinitrophenyl, m.  $210^{\circ}$ .<sup>188</sup>

*p*-Aminosulfonylphenyl, m. 214°.75  
 2-Hydroxy-4-aminophenyl, m. 216°.34  
*p*-Salicyl, m. 222°,455b 220°.458  
 3-Hydroxy-4-carbethoxyphenyl, m. 188°.34  
*p*-Tolyl, m. 150°.602  
 Benzyl, m. 175°,147 155°,230c 152°.135  
*p*-Chlorobenzyl, m. 162°.135  
*p*-Hydroxybenzyl, m. 180°.230c  
*p*-Methoxybenzyl, m. 143°,230c 142°.135  
*p*-Benzyloxybenzyl, m. 161°.135  
*p*-Dimethylaminobenzyl, m. 174°,230c 144°.135  
*p*-Acetaminobenzyl, m. 215° dec.135  
 Carboxybenzyl, *m*, m. 185°; *p*, m. 220°.135  
 $\alpha$ -Carboxybenzyl, m. 254°.135  
*p*-Sulfamylbenzyl, m. 204° dec.457  
*p*-Ethylsulfonylbenzyl, m. 155°,230c 153°.135  
*p*-Dimethylaminosulfonylbenzyl, m. 285°.135  
*p*-Methylbenzyl, m. 120°.135  
 Naphthyl,  $\alpha$  m. 209° dec.;154c  $\beta$  202°,569 204°.575

### *Acyl and Sulfonyl*

Benzoylaminoacetyl, m. 190°.357  
 Propionyl, m. 118°.358  
 Butyryl, m. 77°.358  
 Carbethoxy, m. 184°.166  
 Carbamoyl, m. 219° dec.461  
 Benzoyl, m. 200°,358 198°,357 163–5°.224a  
 Thiobenzoyl, m. 219°,251 picrate 268–71° dec.283  
 Hydroxybenzoyl, m. *o* 202°,357 201° dec.;461 *p* 215°,357 212° dec.461  
*p*-Acetamidobenzoyl, m. 231° dec.;461 221° dec.177b  
 Nitrobenzoyl, m. *o* 210°;357 *p* 215°,358 214°.357  
*p*-Methylsulfonylbenzoyl, m. 234°.452  
 2-Furoyl, m. 205°,505 203°.332  
 Nicotinoyl·HCl, m. 211°.177a  
 Isonicotinoyl, m. 294° dec.;509a 231°.504  
*p*-Aminobenzenesulfonyl, m. 223° dec.457



## MELTING POINTS OF SOME 2-DERIVATIVES



- Methyl, m.  $184^\circ$ ,<sup>194a</sup>  $184^\circ$  dec.,<sup>83a</sup>  $174-6^\circ$ ,<sup>303</sup> H.I., m.  $140^\circ$ .<sup>83c</sup>  
 2,3,4,6-Tetraacetyl- $\beta$ -D-glucosyl, m.  $170^\circ$ ;  $[\alpha]_D$   $29^\circ$ .<sup>256</sup>  
 Phenyl, m.  $153^\circ$ ; <sup>601</sup> picrate, m.  $145^\circ$ .<sup>607</sup>  
 Benzyl, m.  $171^\circ$ ,<sup>230c, 233</sup>  $155^\circ$ .<sup>83a</sup>  
*p*-Methoxybenzyl, m.  $193^\circ$ .<sup>230c</sup>  
*p*-Isopropylbenzyl, m.  $131^\circ$ .<sup>135</sup>  
 3,4-Dichlorobenzyl, m.  $136^\circ$ .<sup>135</sup>  
 3,4-Dimethoxybenzyl, m.  $159^\circ$ .<sup>135</sup>  
 3,4-Dimethylbenzyl, m.  $120^\circ$ .<sup>135</sup>  
*p*-Tolyl, m.  $213-5^\circ$ .<sup>483</sup>  
*p*-Acetamidotolyl, m.  $215-7^\circ$ .<sup>75</sup>  
 $\alpha$ -Phenethyl, m.  $157^\circ$ ,<sup>135</sup>  $156^\circ$ .<sup>230c</sup>  
 $\alpha$ -(*p*-Methoxyphenyl)ethyl, m.  $150^\circ$ .<sup>230c</sup>  
 $\alpha$ -Carboxy- $\beta$ -phenylethyl, m.  $198^\circ$ .<sup>83c</sup>  
 $\alpha,\beta$ -Diphenylethyl, m.  $159^\circ$ .<sup>135</sup>  
 $\alpha$ -Phenylpropyl, m.  $146^\circ$ .<sup>135</sup>  
*p*-Thiosemicarbazidophenylsulfonylguanidine, m.  $237^\circ$  dec.<sup>457</sup>  
 2-(*p*-Thiosemicarbazidophenylsulfonamidopyridine), m.  $230^\circ$  dec.<sup>457</sup>  
 2-(*p*-Thiosemicarbazidophenylsulfonamidethiazole), m.  $230^\circ$ .<sup>457</sup>

*Heterocyclic*

- 3-Pyridyl, m.  $224^\circ$ .<sup>177b</sup>  
 6-Methyl-3-pyridazinyl, m.  $137^\circ$ .<sup>428</sup>  
 4-Methyl-2-pyrimidyl, m.  $255^\circ$  dec.<sup>427</sup>

*Acyl*

- Formyl, m.  $174^\circ$ .<sup>190</sup>  
 Acetyl, m.  $167.5^\circ$ ,<sup>26</sup>  $157^\circ$ ,<sup>501</sup>  $92-5^\circ$ ,<sup>357</sup>  $90^\circ$ .<sup>358</sup>  
 Phenylacetyl, m.  $223^\circ$ ,<sup>358</sup>  $184^\circ$ ,<sup>357</sup>  $178^\circ$ .<sup>461</sup>

## MELTING POINTS OF SOME 4-DERIVATIVES



- Methyl, m.  $137^\circ$ .<sup>293</sup>  
 Ethyl, m.  $84^\circ$ .<sup>161a, 293</sup>

$\alpha$ -(8-Methyl-2-phenanthryl)-*i*-propyl, m. 189°. <sup>152</sup>  
 Butyl, m. 74.5°. <sup>293</sup>  
 Heptyl, m. 55°, <sup>24</sup> 54°. <sup>293</sup>  
 Allyl, m. 99°, <sup>386</sup> 97°. <sup>293</sup>  
 Phenyl, m. 141°, <sup>134</sup> 140°. <sup>385, 467d</sup>  
 Chlorophenyl, *m*, m. 115°; <sup>73b</sup> *p*, 191°, <sup>73b</sup> 180°, <sup>293</sup> 130°. <sup>203</sup>  
*p*-Bromophenyl, m. 189°. <sup>203</sup>  
*p*-Fluorophenyl, m. 189°. <sup>73b</sup>  
*p*-Hydroxyphenyl, m. 171°. <sup>293</sup>  
 Anisyl, *o*, m. 161°, <sup>134</sup> 156°, <sup>167</sup> 154°; <sup>293</sup> *p*, 151°. <sup>293</sup>  
*p*-Ethoxyphenyl, m. 145°. <sup>73b</sup>  
*p*-Carboxyphenyl, m. above 275°. <sup>134</sup>  
 Benzyl, m. 150–2° dec., <sup>498</sup> 130–5°, <sup>293</sup> 130°. <sup>24</sup>  
*p*-Methoxybenzyl, m. 130°. <sup>230c</sup>  
*p*-Sulfamylbenzyl, m. 181°. <sup>134</sup>  
*p*-*i*-Propylbenzyl, m. 96°. <sup>230c</sup>  
*p*-Butylbenzyl, m. 70°. <sup>230c</sup>  
*p*-*i*-Butylbenzyl, m. 68°. <sup>230c</sup>  
*p*-*s*-Butylbenzyl, m. 52°. <sup>230c</sup>  
 Tollyl, *o*, m. 148°, <sup>293, 167</sup> 147°; <sup>467d</sup> *m*, 110°, <sup>467d</sup> 108°; <sup>73b</sup> *p*, 177°, <sup>203</sup>  
     142°, <sup>164</sup> 138°, <sup>467d</sup> 137°, <sup>293</sup> 135°. <sup>535</sup>  
 Benzohydryl, m. 144°. <sup>527</sup>  
*p*-Ethylphenyl, m. 131°. <sup>73b</sup>  
 Naphthyl,  $\alpha$ , m. 139°, <sup>535</sup> 184°; <sup>203</sup>  $\beta$ , 178°. <sup>73b</sup>  
 2-Pyridyl, m. 194°. <sup>134</sup>  
 Propionyl, m. 150°. <sup>500</sup>

## MELTING POINTS OF SOME 1,1-DERIVATIVES

RR'NNHCSNH<sub>2</sub>

| R | R' |
|---|----|
|---|----|

|                          |            |                             |
|--------------------------|------------|-----------------------------|
| Methyl,                  | Phenyl,    | m. 187°. <sup>568</sup>     |
| Carbethoxy,              | Phenyl,    | m. 221°. <sup>514</sup>     |
| Carbamyl,                | Phenyl,    | m. 235° dec. <sup>601</sup> |
| Phenyl,                  | Phenyl,    | m. 202°. <sup>620</sup>     |
|                          | Benzoyl,   | m. 185°. <sup>442a</sup>    |
| Benzyl,                  | Acetyl,    | m. 189°. <sup>135</sup>     |
|                          | Succinoyl, | m. 148–50°. <sup>135</sup>  |
| <i>p</i> -Methoxybenzyl, | Acetyl,    | m. 200°. <sup>135</sup>     |
|                          | Succinoyl, | m. 191°. <sup>135</sup>     |

$\beta$ -Isoquinolinoyl,  $\beta$ -Isoquinolinoyl, m. 248°; HAc, 200–225°; Ag salt, 243° dec.<sup>212</sup>

# MELTING POINTS OF SOME 1,4-DERIVATIVES



R      R'

- Methyl, Phenyl, m. 143°.<sup>56</sup>  
*i*-Propyl, Phenyl, m. 141.5°.<sup>298b</sup>  
 Allyl, Phenyl, m. 123°.<sup>175</sup>  
 Cyclohexyl, (1 or 2) Phenyl, m. 143.5°.<sup>580</sup>  
 3-Methylcyclohexyl, (1 or 2), Phenyl, m. 136°.<sup>588</sup> 135°;  $[\alpha]_D$  -17.66°.<sup>579</sup>  
 Menthyl, Phenyl, m. 160°;  $[\alpha]_D$  -49.1°.<sup>579</sup>  
 $\beta$ -Thujyl, Phenyl, m. 135°.<sup>579</sup>  
 Phenyl, Methyl, m. 170°.<sup>62</sup> 164°.<sup>585</sup>  
     *d*-*s*-Butyl, m. 135°.<sup>46</sup>  
     Hexyl, m. 142°.<sup>297</sup>  
     CHMeC<sub>9</sub>H<sub>19</sub>, m. 109°.<sup>603</sup>  
     Allyl, m. 119°.<sup>175, 421, 554</sup> 118°.<sup>18</sup>  
     CH<sub>2</sub>:CClCH<sub>2</sub>, m. 108°.<sup>554</sup>  
     Phenyl, m. 178°.<sup>391</sup> 177°.<sup>143a, 565, 624</sup> 176°.<sup>64, 585</sup>  
     Chlorophenyl, *m*, m. 168°; *p*, m. 165°.<sup>585</sup>  
     *p*-Bromophenyl, m. 180°.<sup>391</sup>  
     *p*-Anisyl, m. 169°.<sup>73b</sup>  
     Ethoxyphenyl, *o*, m. 185°; *p*, 182°.<sup>73b</sup>  
     *p*-Isoamyloxyphenyl, m. 179°.<sup>73b</sup>  
     *m*-Nitrophenyl, m. 172°.<sup>451</sup>  
     Benzyl, m. 162°.<sup>532, 554</sup> 158°.<sup>46</sup>  
     Benzohydryl, m. 198° dec.<sup>625</sup>  
     Tolyl, *o*, m. 171°.<sup>391</sup> 164°.<sup>585</sup> 163° dec.,<sup>554</sup> 156°.<sup>624</sup> 153°.<sup>64</sup>  
         105°;<sup>18</sup> *m*, 174°;<sup>391</sup> *p*, 186°.<sup>73b</sup> 176°.<sup>585</sup> 174°.<sup>554</sup>  
         165°.<sup>624</sup> 128°.<sup>18</sup>  
     Xylol, 2,3, m. 198°;<sup>73b</sup> 2,4, 173°;<sup>73b</sup> 159°;<sup>585</sup> 2,6, 210°  
         dec.<sup>57c</sup>  
     2(*p*-Cymyl), m. 168.5°.<sup>286</sup>  
     *p*-Butylphenyl, m. 178°.<sup>73b</sup>  
     Naphthyl,  $\alpha$ , m. 193°.<sup>391</sup> 183° dec.,<sup>554</sup> 178°;<sup>73b</sup>  $\beta$ , 203°;<sup>73b</sup>  
         191°.<sup>554</sup> 155°.<sup>18</sup>  
      $\beta$ -Methoxy- $\alpha$ -Methylacryloyl, m. 181° dec.<sup>426</sup>

| R   | R'  |
|---|---|
| Chlorophenyl,   | Methyl, <i>o</i> , m. 147°; <sup>585</sup> <i>m</i> , 177°; <sup>63</sup> 171°. <sup>585</sup><br>Ethyl, <i>p</i> , m. 175°. <sup>585</sup><br>Phenyl, <i>o</i> , m. 156°; <sup>585</sup> <i>m</i> , 153° dec.; <sup>63</sup> 139°; <sup>572</sup><br><i>p</i> , 177°. <sup>585</sup><br><i>m</i> -Chlorophenyl, <i>m</i> , m. 142°. <sup>585</sup><br><i>p</i> -Chlorophenyl, <i>p</i> , m. 160°. <sup>585</sup>   |
| Bromophenyl,  | Methyl, <i>m</i> , m. 175° dec.; <sup>63</sup> <i>p</i> , 199°. <sup>585</sup><br>Ethyl, <i>p</i> , m. 190°. <sup>585</sup><br>Phenyl, <i>m</i> , m. 160°; <sup>63</sup> <i>p</i> , 200°. <sup>585</sup><br>$\alpha$ -Naphthyl, <i>p</i> , m. 185°. <sup>585</sup><br>$\beta$ -Naphthyl, <i>p</i> , m. 202°. <sup>585</sup>   |
| 2,4,6-Tribromophenyl,   | Methyl, m. 206°. <sup>585</sup><br>Phenyl, m. 203°. <sup>585</sup><br><i>p</i> -Tolyl, m. 201°. <sup>585</sup>  |
| <i>o</i> -Methoxyphenyl,  | Methyl, m. 153°. <sup>585</sup><br>Phenyl, m. 152°; <sup>63</sup> 151°. <sup>585</sup>  |
| <i>p</i> -Ethoxyphenyl,   | <i>p</i> -Ethoxyphenyl, m. 156–8°; <sup>171b, 170</sup> 156°. <sup>3</sup><br><i>p</i> -Butoxyphenyl, m. 128°. <sup>3</sup>   |
| <i>p</i> -Butoxyphenyl,   | <i>p</i> -Ethoxyphenyl, m. 143°. <sup>3</sup><br><i>p</i> -Butoxyphenyl, m. 149°. <sup>3</sup>  |
| <i>p</i> -Crotyloxyphenyl,  | <i>p</i> -Butoxyphenyl, m. 135–7°. <sup>3</sup>   |
| Nitrophenyl,  | Methyl, <i>o</i> , m. 202°; <i>m</i> , 177°; <i>p</i> , 233°. <sup>585</sup><br>Ethyl, <i>o</i> , m. 168°. <sup>585</sup><br>Allyl, <i>o</i> , m. 166°; <i>m</i> , 120°; <i>p</i> , 188°. <sup>421</sup><br>Phenyl, <i>o</i> , m. 186°; <sup>585</sup> <i>m</i> , 164°; <sup>63</sup> <i>p</i> , 220°. <sup>585</sup>   |
| 2,4-O <sub>2</sub> N(Br)C <sub>6</sub> H <sub>3</sub> ,             | Phenyl, m. 160–64°. <sup>524</sup>  |
| 2,4-(O <sub>2</sub> N) <sub>2</sub> C <sub>6</sub> H <sub>3</sub> , | Phenyl, m. 187°; <sup>549</sup> 186°. <sup>605</sup>  |
| <i>m</i> -HOCC <sub>6</sub> H <sub>4</sub> ,                        | Phenyl, m. 205° dec. <sup>606</sup>   |
| Benzyl,   | Phenyl, m. 155°. <sup>62</sup>  |
| Benzohydryl, (1 or 2),  | Phenyl, m. 164°. <sup>551</sup>   |
| $\alpha$ -Carboxy- $\beta$ -phenylethyl,                            | Benzyl, m. 167°. <sup>83c</sup>   |
| Tolyl,  | Methyl, <i>o</i> , m. 159°; <sup>585</sup> <i>m</i> , m. 163°; <sup>63</sup> <i>p</i> , m. 170°. <sup>585</sup><br>Ethyl, <i>o</i> , m. 131°. <sup>554, 585</sup><br>Allyl, <i>o</i> , m. 105°; <sup>18</sup> <i>p</i> , m. 128°. <sup>18</sup><br>Phenyl, <i>o</i> , m. 147°; <sup>63, 585</sup> 146°; <sup>554</sup> <i>m</i> , m. 157°; <sup>63</sup> <i>p</i> , m. 175°; <sup>585</sup> 173°; <sup>297</sup> 172°. <sup>554</sup><br><i>o</i> -Tolyl, <i>o</i> , m. 149° dec.; <sup>554</sup> <i>m</i> , m. 148°; <sup>63</sup> <i>p</i> , m. 163° dec. <sup>554, 585</sup><br><i>p</i> -Tolyl, <i>o</i> , m. 142°; <sup>554</sup> <i>m</i> , m. 146°; <sup>63</sup> <i>p</i> , m. 154° dec. <sup>585</sup> |

| R   | R'   |
|---|--|
| Tolyl,  | <i>p</i> -Chlorophenyl, <i>p</i> , m. 146.5°. <sup>585</sup>                                 |
|   | Benzyl, <i>p</i> , m. 156°. <sup>530</sup>   |
|   | 2,4-Xylyl, <i>p</i> , m. 170°. <sup>585</sup>  |
|   | α-Naphthyl, <i>o</i> , m. 176°; <i>p</i> , m. 184°. <sup>585</sup>                           |
|   | β-Naphthyl, <i>p</i> , m. 184°. <sup>585</sup>   |
| 2,4-BrMeC <sub>6</sub> H <sub>3</sub> ,                 | Allyl, m. 136.5°. <sup>573</sup>   |
|   | Phenyl, m. 142°. <sup>573</sup>  |
| 2,4-O <sub>2</sub> N(Me)C <sub>6</sub> H <sub>3</sub> , | Allyl, m. 168–70°. <sup>604</sup>  |
|   | Phenyl, m. 188°. <sup>604</sup>  |
| Xylyl, Methyl (2,4),                                    | m. 180°. <sup>585</sup>  |
|   | Phenyl (2,4), m. 149°; <sup>585</sup> (2,6), m. 182° dec. <sup>57a</sup>                     |
| <i>p</i> -Biphenyl,                                     | (1 or 2), Phenyl, m. 182°. <sup>595</sup>  |
| Naphthyl, Methyl,                                       | α, m. 195°; <sup>585</sup> β, m. 212°, <sup>62</sup> 209°. <sup>554</sup>                    |
|   | Ethyl, α, m. 149°; <sup>585</sup> β, m. 169°. <sup>554</sup>                                 |
|   | Allyl, β, m. 155°. <sup>18</sup>   |
|   | Phenyl, α, m. 220°; <sup>585</sup> β, m. 202°, <sup>154c, 585</sup> 196° dec. <sup>57c</sup> |
|   | <i>o</i> -Tolyl, β, m. 192°. <sup>585</sup>  |
|   | <i>p</i> -Tolyl, α, m. 169°; β, m. 195°. <sup>585</sup>                                      |
|   | α-Naphthyl, α, m. 192°; β, m. 207°. <sup>585</sup>   |
|   | β-Naphthyl, α, m. 179°; β, m. 187°. <sup>585</sup>   |
| 7,2-HOC <sub>10</sub> H <sub>6</sub> ,                  | Phenyl, m. 183°. <sup>563</sup>  |
| Benzothiazolyl,   | Phenyl, m. 194°. <sup>182</sup>  |
|   | <i>p</i> -Tolyl, m. 194°. <sup>182</sup>   |
|   | α-Naphthyl, m. 110°. <sup>182</sup>  |
|   | <i>p</i> -Phenylazophenyl, m. 155°. <sup>182</sup>   |

### Acyl Compounds

|   |  |
|---|--|
| Carbethoxy,                               | Phenyl, m. 141°. <sup>166</sup>                    |
| Acetyl,                                   | Phenyl, m. 172°. <sup>262</sup>                    |
|   | Benzoyl, m. 188° dec. <sup>442b</sup>              |
| ( <i>p</i> -Phenethylamino)acetyl,        | Phenyl, m. 172°. <sup>466</sup>                    |
|   | <i>p</i> -Chlorophenyl, m. 189°. <sup>466</sup>    |
| ( <i>p</i> -Cyclohexylphenylamino)acetyl, | Phenyl, m. 196°. <sup>466</sup>                    |
| Phenylacetyl,                             | Benzoyl, m. 170°. <sup>442b</sup>                  |
| Di- <i>p</i> -Chlorophenylacetyl,         | β,β-Diphenylacrylyl, m. 205°. <sup>296</sup>       |
|   | 3,4,5-Trimethoxybenzoyl, m. 185.8°. <sup>296</sup> |
|   | 2-Furoyl, m. 202.8°. <sup>296</sup>                |
| Cinnamoyl,                                | Phenyl, m. 146°. <sup>594</sup>                    |
| Butyryl,                                  | Butyryl, m. 120°. <sup>500</sup>                   |

| R  | R'   |
|--|--|
| Lauroyl, <i>p</i> -Isoamyloxyphenyl, m.              | 149°.73a   |
| Palmitoyl, <i>p</i> -Ethoxyphenyl, m.                | 143°.73a   |
| Coumariloyl, m.                                      | 188°.73a   |
| Carbethoxy, Cyclohexyl, m.                           | 124°.467e  |
| Phenyl, m.   | 146°.467e  |
| Chlorophenyl, <i>m</i> , m.                          | 143°; <i>p</i> , 155°.467e                           |
| <i>p</i> -Bromophenyl, m.                            | 153°.467e  |
| Anisyl, <i>o</i> , m.                                | 154°; <i>p</i> , m. 156°.467e                        |
| Benzyl, m.   | 127°.467e  |
| Tolyl, <i>o</i> , m.                                 | 83-5°; <i>m</i> , m. 120.5°; <i>p</i> , m. 157°.467e |
| Xylyl (2,3), m.                                      | 151°; (2,4), m. 114°; (2,5), m. 107°.467e            |
| Carbamoyl, Phenylacetyl, m.                          | 189°.442c  |
| Benzoyl, m.  | 209°.442c  |
| Benzoyl, Methyl, m.                                  | 195°.230a  |
| <i>i</i> -Propyl, m.                                 | 175°.230a  |
| Phenyl, m.   | 167°.62 166°.230a 164-6°.442a 162°.311               |
| Acetyl, m.   | 129° dec.442b  |
| <i>p</i> -Chlorobenzoyl, <i>p</i> -Chlorobenzoyl, m. | 235° dec.230a  |
| Phenylacetyl, m.                                     | 167.2°.296   |
| Phenylpropionyl, m.                                  | 171.6°.296   |
| <i>o</i> -Hydroxybenzoyl, <i>p</i> -Fluorophenyl, m. | 210°.73a   |
| <i>p</i> -Anisyl, m.                                 | 214°.73a   |
| <i>p</i> -Ethoxyphenyl, m.                           | 212°.73a   |
| <i>p</i> -( <i>i</i> -Amyloxyphenyl), m.             | 209°.73a   |
| <i>p</i> -Tolyl, m.                                  | 218°.73a   |
| Xylyl (2,3), m.                                      | 189°; (2,4), m. 201°.73a                             |
| <i>o</i> -Biphenyl, m.                               | 188°.73a   |
| Naphthyl, $\alpha$ , m.                              | 215°, $\beta$ , 208°.73a                             |
| <i>p</i> -Methoxybenzoyl, Methyl, m.                 | 210°.230a  |
| Phenyl, m.   | 184°.230a  |
| <i>p</i> -Methoxybenzoyl, m.                         | 210°.230a  |
| 5-Bromo-2-formylbenzoyl, <i>p</i> -Anisyl, m.        | 216°.73a   |
| <i>p</i> -Ethoxyphenyl, m.                           | 212°.73a   |
| 2,4-Xylyl, m.  | 192°.73a   |
| <i>o</i> -Biphenyl, m.                               | 189°.73a   |
| Naphthyl, $\alpha$ , m.                              | 232°; $\beta$ , m. 204°.73a                          |

| R                                    | R'  |
|--------------------------------------|---|
| 3,5-Dichloro-2-hydroxybenzoyl,       | <i>p</i> -Anisyl, m. 195°. <sup>73a</sup><br><i>p</i> -Ethoxyphenyl, m. 184°. <sup>73a</sup><br><i>p</i> -Tolyl, m. 197°. <sup>73a</sup><br>$\alpha$ -Naphthyl, m. 190°. <sup>73a</sup>   |
| 3,5-Dibromo-2-hydroxybenzoyl,        | <i>p</i> -Ethoxyphenyl, m. 178°. <sup>73a</sup>   |
| Yohimbic, Phenyl,                    | m. 299–303°. <sup>240b</sup>  |
| Anilinothiocarbonyl,                 | <i>p</i> -Chlorophenyl, m. 216°. <sup>73b</sup><br><i>p</i> -Bromophenyl, m. 219°. <sup>73b</sup><br><i>p</i> -Fluorophenyl, m. 221°. <sup>73b</sup><br><i>p</i> -Anisyl, m. 211°. <sup>73b</sup><br><i>p</i> -Ethoxyphenyl, m. 219°. <sup>73b</sup><br><i>p</i> -( <i>i</i> -Amyloxyphenyl), m. 209°. <sup>73b</sup><br><i>p</i> -Tolyl, m. 210°. <sup>73b</sup><br>Xylyl, (2,3), m. 198°; (2,4), m. 205°. <sup>73b</sup><br>Naphthyl, $\alpha$ , m. 191°; $\beta$ , m. 201°. <sup>73b</sup> |
| <i>p</i> -Chloroanilinothiocarbonyl, | <i>p</i> -Chlorophenyl, m. 232°. <sup>73b</sup><br><i>p</i> -Bromophenyl, m. 234°. <sup>73b</sup><br><i>p</i> -Anisyl, m. 228°. <sup>73b</sup><br><i>p</i> -Ethoxyphenyl, m. 230°. <sup>73b</sup><br><i>p</i> -Tolyl, m. 235°. <sup>73b</sup><br>Naphthyl, $\alpha$ , m. 212°; $\beta$ , 221°. <sup>73b</sup>   |
| <i>p</i> -Bromoanilinothiocarbonyl,  | <i>p</i> -Bromophenyl, m. 234°. <sup>73b</sup><br><i>p</i> -Anisyl, m. 231°. <sup>73b</sup><br><i>p</i> -Ethoxyphenyl, m. 228°. <sup>73b</sup><br><i>p</i> -Tolyl, m. 233°. <sup>73b</sup><br>Naphthyl, $\alpha$ , m. 215°; $\beta$ , 223°. <sup>73b</sup>  |
| <i>p</i> -Fluoroanilinothiocarbonyl, | <i>p</i> -Chlorophenyl, m. 234°. <sup>73b</sup><br><i>p</i> -Bromophenyl, m. 238°. <sup>73b</sup><br><i>p</i> -Fluorophenyl, m. 230°. <sup>73b</sup><br><i>p</i> -Anisyl, m. 229°. <sup>73b</sup><br><i>p</i> -Ethoxyphenyl, m. 229°. <sup>73b</sup><br>Tolyl, <i>m</i> , m. 219°; <i>p</i> , m. 231°. <sup>73b</sup><br>Naphthyl, $\alpha$ , m. 206°; $\beta$ , 220°. <sup>73b</sup>   |
| <i>p</i> -Anisidinothiocarbonyl,     | <i>p</i> -Anisyl, m. 237°. <sup>73b</sup><br><i>p</i> -Ethoxyphenyl, m. 227°. <sup>73b</sup><br>Naphthyl, $\alpha$ , m. 204°; $\beta$ , m. 225°. <sup>73b</sup>   |
| <i>p</i> -Toluidinothiocarbonyl,     | <i>p</i> -Anisyl, m. 225°. <sup>73b</sup><br><i>p</i> -Ethoxyphenyl, m. 231°. <sup>73b</sup><br><i>p</i> -Tolyl, m. 227°. <sup>73b</sup><br>Naphthyl, $\alpha$ , m. 204°; $\beta$ , m. 219°. <sup>73b</sup>   |

| R                                  | R'   |
|------------------------------------|--|
| <i>p</i> -Phenetidinothiocarbonyl, | <i>p</i> -Ethoxyphenyl, m. 239°. <sup>73b</sup>                      |
|                                    | Naphthyl, $\alpha$ , m. 202°; $\beta$ , m. 227°. <sup>73b</sup>      |
| Nicotinoyl,                        | <i>p</i> -Anisyl, m. 169°. <sup>73a</sup>                            |
|                                    | <i>p</i> -Ethoxyphenyl, m. 168°. <sup>73a</sup>                      |
|                                    | <i>p</i> -Isoamyloxyphenyl, m. 217°. <sup>73a</sup>                  |
|                                    | 2,4-Xylyl, m. 170°. <sup>73a</sup>                                   |
|                                    | <i>o</i> -Biphenylyl, m. 195°. <sup>73a</sup>                        |
|                                    | Naphthyl, $\alpha$ , m. 208°; $\beta$ , m. 210°. <sup>73a</sup>      |
| Nicotinoyloxide,                   | Allyl, m. 191°. <sup>126</sup>                                       |
|                                    | Phenyl, m. 181°. <sup>126</sup>                                      |
|                                    | <i>m</i> -Nitrophenyl, m. 219°. <sup>126</sup>                       |
|                                    | Benzyl, m. 199°. <sup>126</sup>                                      |
|                                    | <i>p</i> -Tolyl, m. 199°. <sup>126</sup>                             |
| Isonicotinoyl,                     | 2,3,4,6-Tetraacetyl- $\beta$ -D-glucosyl, m. 167°. <sup>321</sup>    |
|                                    | Allyl, m. 179°. <sup>505</sup>                                       |
|                                    | <i>p</i> -Fluorophenyl, m. 217°. <sup>73a</sup>                      |
|                                    | <i>p</i> -Anisyl, m. 197°. <sup>73a</sup>                            |
|                                    | Ethoxyphenyl, <i>o</i> , m. 203°; <i>p</i> , m. 202°. <sup>73a</sup> |
|                                    | <i>p</i> -( <i>i</i> -Amyloxyphenyl), m. 217°. <sup>73a</sup>        |
|                                    | <i>p</i> -Tolyl, m. 206°. <sup>73a</sup>                             |
|                                    | Xylyl (2,3), m. 214°; (2,4), m. 200°. <sup>73a</sup>                 |
|                                    | <i>o</i> -Biphenylyl, m. 203°. <sup>73a</sup>                        |
|                                    | Naphthyl, $\alpha$ , m. 232°; $\beta$ , m. 208°. <sup>73a</sup>      |
|                                    | Phenylacetyl, m. 139.6°. <sup>296</sup>                              |
|                                    | Propionyl, m. 162.2°. <sup>296</sup>                                 |
|                                    | $\beta$ -Phenylpropionyl, m. 182.4°. <sup>296</sup>                  |
|                                    | Butyryl, m. 163°. <sup>296</sup>                                     |
| Isonicotinoyl,                     | <i>t</i> -Butyryl, m. 131.4°. <sup>296</sup>                         |
|                                    | $\alpha,\alpha$ -Dimethylbutyryl, m. 145.4°. <sup>296</sup>          |
|                                    | $\gamma$ -Phenylbutyryl, m. 167.4°. <sup>296</sup>                   |
|                                    | Valeryl, m. 164.8°. <sup>296</sup>                                   |
|                                    | Isovaleryl, m. 163°. <sup>296</sup>                                  |
|                                    | Pivaloyl, m. 133°. <sup>296</sup>                                    |
|                                    | $\alpha$ -Methylvaleryl, m. 160.8°. <sup>296</sup>                   |
|                                    | $\alpha,\alpha$ -Dimethylvaleryl, m. 116°. <sup>296</sup>            |
|                                    | Caproyl, m. 133.8°. <sup>296</sup>                                   |
|                                    | $\alpha$ -Ethylcaproyl, m. 168.2°. <sup>296</sup>                    |
|                                    | Heptoyl, m. 155.8°. <sup>296</sup>                                   |
|                                    | Caprylyl, m. 124°. <sup>296</sup>                                    |
|                                    | Pelargonyl, m. 103°. <sup>296</sup>                                  |



| R                                   | R'   |
|-------------------------------------|--|
| Isonicotinoyl,                      | Capryl, m. 117.8°. <sup>296</sup>  |
|                                     | Undecyloyl, m. 127°. <sup>296</sup>  |
|                                     | Lauroyl, m. 124°. <sup>296</sup>   |
|                                     | Myristoyl, m. 112.8°. <sup>296</sup>   |
|                                     | Palmitoyl, m. 126.8°. <sup>296</sup>   |
|                                     | Stearoyl, m. 129°. <sup>296</sup>  |
|                                     | $\beta$ -Ethylacryloyl, m. 181°. <sup>296</sup>  |
|                                     | $\beta$ -Propylacryloyl, m. 156°. <sup>296</sup>   |
|                                     | $\beta$ -( <i>i</i> -Propylacryloyl), m. 182°. <sup>296</sup>                                  |
|                                     | $\beta$ -Butylacryloyl, m. 134.8°. <sup>296</sup>  |
|                                     | $\beta$ -Amylacryloyl, m. 140.2°. <sup>296</sup>   |
|                                     | $\beta$ -Hexylacryloyl, m. 139.6°. <sup>296</sup>  |
|                                     | $\beta$ -Heptylacryloyl, m. 99°. <sup>296</sup>  |
|                                     | $\beta$ -Octylacryloyl, m. 125°. <sup>296</sup>  |
|                                     | $\beta$ -Decylacryloyl, m. 139.6°. <sup>296</sup>  |
|                                     | $\alpha$ -Ethyl- $\beta$ -propylacryloyl, m. 152.6°. <sup>296</sup>                            |
|                                     | $\beta,\beta$ -Diphenylacryloyl, m. 212°. <sup>296</sup>                                       |
|                                     | $\beta$ -(3-Furylacryloyl), m. 264.5° dec. <sup>296</sup>                                      |
|                                     | $\gamma$ -Methyl- $\gamma$ -propylcrotonyl, m. 141.2°. <sup>296</sup>                          |
|                                     | Sorboyl, m. 178°. <sup>296</sup>   |
|                                     | Oleoyl, m. 115.8°. <sup>296</sup>  |
|                                     | C <sub>8</sub> H <sub>17</sub> ≡C(CH <sub>2</sub> ) <sub>7</sub> CO, m. 105.8°. <sup>296</sup> |
|                                     | Tricyclenoyl, m. 85.8°. <sup>296</sup>   |
|                                     | <i>p</i> -Anisoyl, m. 177°. <sup>296</sup>   |
|                                     | 3,4,5-Trimethoxybenzoyl, m. 188.2°. <sup>296</sup>   |
|                                     | Furoyl, m. 184°. <sup>296</sup>  |
|                                     | Nicotinoyl, 266°. <sup>296</sup>   |
|                                     | Isonicotinoyl, m. 202°. <sup>296</sup>   |
| <i>p</i> -Aminobenzenesulfonyl,     | Methyl, m. 242–5° dec. <sup>194b</sup>   |
|                                     | Allyl, m. 176°. <sup>429.5</sup>   |
|                                     | Phenyl, m. 195°. <sup>429.5</sup>  |
| <i>p</i> -Acetaminobenzenesulfonyl, | Methyl, m. 225–7° dec. <sup>194b</sup>   |
|                                     | Allyl, m. 198°. <sup>429.5</sup>   |
|                                     | Phenyl, m. 213°. <sup>429.5</sup>  |
|                                     | $\alpha$ -Naphthyl, m. 200°. <sup>429.5</sup>  |
| Phenyl, Acetyl,                     | m. 179°. <sup>554</sup>  |
|                                     | Propionyl, m. about 156° dec. <sup>554</sup>   |
|                                     | Benzoyl, m. 137°, <sup>626</sup> 136°. <sup>253</sup>  |
|                                     | Carbomethoxy, m. 180°. <sup>556</sup>  |

| R                       | R'                       |                      |
|-------------------------|--------------------------|----------------------|
| Phenyl, Carboethoxy, m. | 146.5°. <sup>556</sup>   |                      |
|                         | Methylphenylcarbamyl, m. | 121°. <sup>554</sup> |
|                         | Diphenylcarbamyl, m.     | 153°. <sup>554</sup> |

## MELTING POINTS OF SOME 2,4-DERIVATIVES



| R   | R'   |
|---|--|
| Methyl, Methyl, m.                                | 138°. <sup>471, 62, 311</sup>  |
|   | Ethyl, m. 84°. <sup>311</sup>  |
|   | <i>i</i> -Propyl, m. 107°. <sup>230c</sup>   |
|   | Butyl, m. 50°. <sup>230c</sup>   |
|   | Allyl, m. 57°. <sup>62</sup>   |
|   | Phenyl, m. 115°. <sup>582</sup>  |
| Ethyl, Phenyl, m.                                 | 110°. <sup>143b</sup>  |
| Pr <sub>2</sub> CH, Phenyl, m.                    | 122°. <sup>579</sup>   |
| MeHexCH, Phenyl, m.                               | 116°. <sup>579</sup>   |
| (EtO) <sub>2</sub> CHCH <sub>2</sub> , Phenyl, m. | 98°. <sup>559</sup>  |
| Allyl, Phenyl, m.                                 | 123°. <sup>175</sup>   |
| Phenyl, Methyl, m.                                | 91°, <sup>585</sup> 90°, 89°; <sup>554</sup> HCl, m. 177°. <sup>62</sup>   |
|   | Ethyl, m. 122°. <sup>554</sup>   |
|   | Camphoryl, m. 183°; [α] <sub>D</sub> 59.5° (CHCl <sub>3</sub> ). <sup>560</sup>  |
|   | Phenyl, m. 139°; <sup>585</sup> HCl, m. 170° dec. <sup>57a</sup>   |
|   | Chlorophenyl, <i>m</i> , m. 120°; <i>p</i> , m. 134°, <sup>585</sup> sinters at 158°. <sup>73b</sup>                                 |
|   | <i>p</i> -Bromophenyl, sinters at 165°. <sup>73b</sup>   |
|   | <i>p</i> -Anisyl, sinters at 155°. <sup>73b</sup>  |
|   | <i>p</i> -Ethoxyphenyl, sinters at 150°. <sup>73b</sup>  |
|   | Tolyl, <i>o</i> , m. 134°; <sup>585</sup> <i>p</i> , sinters at 150°. <sup>554, 585</sup>  |
|   | Xylol (2,3), sinters at 172°; <sup>73b</sup> (2,4), m. 145°. <sup>585</sup>  |
|   | Benzyl, m. 116°. <sup>554</sup>  |
|   | Benzohydryl, m. 178°. <sup>625</sup>   |
|   | <i>p</i> -Butylphenyl, sinters at 134°. <sup>73b</sup>   |
|   | β-Naphthyl, sinters at 185°. <sup>73b</sup>  |
| Chlorophenyl, Methyl, <i>m</i> , m.               | 105°. <sup>63</sup>  |
|   | Ethyl, <i>p</i> , m. 138°. <sup>585</sup>  |
|   | Phenyl, <i>o</i> , m. 134°; <sup>572</sup> <i>m</i> , m. 117°; <sup>63</sup> <i>p</i> , m. 150°, <sup>585</sup> 149°. <sup>572</sup> |
|   | <i>p</i> -Chlorophenyl, <i>p</i> , m. 142°. <sup>585</sup>   |

| R   | R'                             |   |
|---|--------------------------------|---|
| Bromophenyl,  | Methyl, <i>p</i> , m.          | 133°.585  |
|   | Ethyl, <i>p</i> , m.           | 146°.585  |
|   | Phenyl, <i>m</i> , m.          | 130°; 63 <i>p</i> , m. 160°.585                     |
|   | β-Naphthyl, <i>p</i> , m.      | 183°.585  |
| Nitrophenyl,  | Methyl, <i>p</i> , m.          | 206°.585  |
|   | Phenyl, <i>m</i> , m.          | 147°, 524 144°; 57c <i>p</i> , m. 198–200°.585      |
| <i>o</i> -Methoxyphenyl,  | Phenyl, m.                     | 141°.63   |
| <i>p</i> -Ethoxyphenyl,   | <i>p</i> -Ethoxyphenyl, m.     | 146°.170  |
| Piperonyl,  | Phenyl, m.                     | 153.5°.100  |
| Tolyl,  | Methyl, <i>m</i> , m.          | 120°; 63 <i>p</i> , m. 119°.585                     |
|   | Phenyl, <i>o</i> , m.          | 124°; <i>m</i> , m. 133°; 63 <i>p</i> , m. 123°.585 |
|   | <i>p</i> -Chlorophenyl, m.     | 145°.585  |
|   | <i>o</i> -Tolyl, <i>m</i> , m. | 121°; HCl, m. 162°; <i>p</i> , m. 131°.554, 585     |
|   | <i>p</i> -Tolyl, <i>m</i> , m. | 131°; 63 <i>p</i> , m. 125°.554, 585                |
|   | 2,4-Xylyl, <i>p</i> , m.       | 152°.585  |
|   | Benzyl, <i>p</i> , m.          | 121°.554  |
|   | β-Naphthyl, <i>p</i> , m.      | 125°.585  |
| 2,6-Xylyl,  | Phenyl, m.                     | 145°.57c  |
| Benzyl,   | Methyl, m.                     | 129°.62   |
|   | Allyl, m.                      | 61°.62  |
|   | Phenyl, m.                     | 145°.62   |
| <i>p</i> -MeC <sub>6</sub> H <sub>4</sub> CH <sub>2</sub> ,           | Phenyl, m.                     | 133°.550  |
| 2,4-Me <sub>2</sub> C <sub>6</sub> H <sub>3</sub> CH <sub>2</sub> ,   | Phenyl, m.                     | 138.5°.100  |
| 2,4,5-Me <sub>3</sub> C <sub>6</sub> H <sub>2</sub> CH <sub>2</sub> , | Phenyl, m.                     | 168°.550  |
| Naphthyl,   | Methyl, β, m.                  | 172° dec.63   |
|   | Phenyl, α, m.                  | 135°; 154c β, m. 185°, 63 184.5°.554                |

## MELTING POINTS OF SOME 1,1,4-DERIVATIVES

## RR'NNHCSNHR''

| R       | R'                            | R''                            |
|---------|-------------------------------|--------------------------------|
| Methyl, | Phenyl,                       | Methyl, m. 161.5°.585          |
|         | Phenyl, <i>s</i> -Butyl, m.   | 112°; 46 <i>D</i> , m. 112°.56 |
|         | Phenyl, Allyl, m.             | 90°.46                         |
|         | Phenyl, Phenyl, m.            | 154°.143a, 624                 |
|         | Acetyl, Phenyl, m.            | 164° dec.225                   |
|         | Propionyl, Phenyl, m.         | 146°, 60 144°, 600 144.5°.225  |
|         | <i>i</i> -Butyryl, Phenyl, m. | 153–5°.225                     |
| Ethyl,  | Phenyl, Phenyl, m.            | 149°.583                       |

| R  | R'   | R'' |
|--|--|-----|
| <i>i</i> -Propyl, <i>i</i> -Propyl, Phenyl, m. 103°. <sup>268</sup>                            |  |     |
|  | Phenyl, Phenyl, m. 116°. <sup>592</sup>  |     |
| Butyl, Butyl, Phenyl, m. 72°. <sup>513</sup>   |  |     |
| <i>i</i> -Butyl, Phenyl, Phenyl, m. 140°. <sup>592</sup>                                       |  |     |
| <i>i</i> -Amyl, Phenyl, Phenyl, m. 220°. <sup>592</sup>  |  |     |
| Allyl, Phenyl, Phenyl, m. 108°. <sup>585</sup> 103°. <sup>591</sup>                            |  |     |
| HOOCCH <sub>2</sub> , Phenyl, Ethyl, m. 155°; Et ester, m. 84°. <sup>533</sup>                 |  |     |
|  | Phenyl, Phenyl, m. 195° dec.; <sup>57c</sup> Et ester, m. 156°. <sup>567</sup> |     |
| Phenyl, Phenyl, Methyl, m. 204° dec. <sup>585</sup>  |  |     |
|  | Phenyl, Phenyl, m. 181°. <sup>585</sup>  |     |
|  | Benzyl, Phenyl, m. 150°. <sup>592</sup>  |     |
|  | Acetyl, Phenyl, m. 160°, again at 261°. <sup>304</sup>                         |     |
|  | Benzoyl, Phenyl, m. 319° dec. <sup>442a</sup>                                  |     |
|  | Phenylcarbamyl, Phenyl, m. 179°. <sup>601</sup>                                |     |
| H <sub>2</sub> NNPhCH <sub>2</sub> CH <sub>2</sub> , Phenyl, Phenyl, m. 164.5°. <sup>589</sup> |  |     |

## SOME 1,2,4-DERIVATIVES

## RNHNR'CSNHR''

| R  | R'   | R'' |
|--|--|-----|
| Methyl, Phenyl, Phenyl, m. 138°. <sup>60</sup>                           |  |     |
| <i>i</i> -Propyl, <i>i</i> -Propyl, Phenyl, m. 130°. <sup>451</sup>      |  |     |
| Phenyl, Methyl, Phenyl, m. 177°. <sup>60</sup> 175°. <sup>583, 585</sup> |  |     |
|  | Phenyl, Phenyl, m. 174°. <sup>585</sup>                      |     |
|  | Phenyl, Acetyl, m. 272.5°. <sup>304</sup>                    |     |
| Formyl, Phenyl, Phenyl, m. 129°. <sup>622</sup>                          |  |     |
| Acetyl, Methyl, Phenyl, m. 154.5°. <sup>225</sup>                        |  |     |
|  | Phenyl, Phenyl, m. 133°. <sup>304</sup> 132°. <sup>622</sup> |     |
| Propionyl, Methyl, Phenyl, m. 170–2° dec. <sup>225</sup>                 |  |     |
|  | <i>i</i> -Propyl, Phenyl, m. 156°. <sup>224b</sup>           |     |
|  | Phenyl, Phenyl, m. 118°. <sup>622</sup>                      |     |
| <i>i</i> -Butyryl, Methyl, Phenyl, m. 175°. <sup>225</sup>               |  |     |
| Phenylacetyl, Phenyl, Phenyl, m. 126°. <sup>622</sup>                    |  |     |
| EtOOCCH <sub>2</sub> CO, Phenyl, Phenyl, m. 141°. <sup>590</sup>         |  |     |
| Benzoyl, Methyl, Methyl, m. 193° dec. <sup>230a</sup>                    |  |     |
|  | Methyl, Phenyl, m. 160°. <sup>225</sup>                      |     |
|  | Phenyl, Phenyl, m. 310°. <sup>593</sup>                      |     |
| <i>p</i> -Anisoyl, Methyl, Methyl, m. 192° dec. <sup>230a</sup>          |  |     |
| Carboxy, Phenyl, Methyl, m. 90°. <sup>534</sup>                          |  |     |

| R               | R'      | R''   |
|-----------------|---------|---|
| Carbethoxy,     | Phenyl, | Phenyl, m. 145°. <sup>531</sup>                               |
| EtSCO,          | Phenyl, | Acetyl, m. 145°. <sup>627</sup>                               |
|                 |         | Benzoyl, m. 148–50°. <sup>627</sup>                           |
| Phenylcarbamyl, | Phenyl, | Phenyl, m. 164°. <sup>60</sup>                                |
|                 | Tolyl,  | Phenyl, <i>o</i> , m. 181°; <i>p</i> , m. 174°. <sup>60</sup> |

## MELTING POINTS OF SOME MISCELLANEOUS THIOSEMICARBAZIDES

|   |                                    |
|---|------------------------------------|
| 4,4-Dimethyl,                           | m. 157°. <sup>249b, 198, 293</sup> |
| 4,4-Diethyl,                            | m. 85°. <sup>249b</sup>            |
| 1-EtSCO-2-Phenyl,                       | m. 173° dec. <sup>627</sup>        |
| 1-Carbamyl-2-Phenyl,                    | m. 213° dec. <sup>601</sup>        |
| 1-Phenyl-4,4-Dipropyl,                  | m. 104°. <sup>64</sup>             |
| 1-Phenyl-4,4-Di- <i>i</i> -Amyl,        | m. 100°. <sup>64</sup>             |
| 1,4-Diphenyl-4-Methyl,                  | m. 142°. <sup>64</sup>             |
| 1-Benzoyl-4,4-Dimethyl,                 | m. 170°. <sup>230a</sup>           |
| 1-( <i>p</i> -Anisoyl)-4,4-Dimethyl,    | m. 167°. <sup>230a</sup>           |
| 1-Isonicotinoyl-4,4-Diethyl,            | m. 186°. <sup>505</sup>            |
| 1,1-Dimethyl-2-(β-Cyanoethyl)-4-Phenyl, | m. 105°. <sup>226</sup>            |
| 1,4-Dimethyl-1,4-Diphenyl,              | m. 113°. <sup>568</sup>            |
| 1,4-Diethyl-1,4-Diphenyl,               | m. 84°. <sup>568</sup>             |

MELTING POINTS OF SOME *bis*-THIOSEMICARBAZIDES

| <i>n</i> | R  |
|----------|--|
| 4        | <i>p</i> -Ethoxyphenyl, m. 212°. <sup>73a</sup>              |
| 4        | <i>p</i> ( <i>i</i> -Amyloxyphenyl), m. 204°. <sup>73a</sup> |
| 7        | <i>p</i> -Bromophenyl, m. 212°. <sup>71</sup>                |
| 7        | <i>p</i> -Anisyl, m. 194°. <sup>71</sup>                     |
| 7        | <i>p</i> -Tolyl, m. 174°. <sup>71</sup>                      |
| 8        | Phenyl, m. 148°. <sup>71</sup>                               |
| 8        | <i>p</i> -Fluorophenyl, m. 178°. <sup>71</sup>               |

MISCELLANEOUS *bis*-THIOSEMICARBAZIDES

|  |                             |
|--|-----------------------------|
| <i>bis</i> -( <i>p</i> -Thiosemicarbazidophenyl)sulfone,                                 | m. 243° dec. <sup>457</sup> |
| S(C <sub>6</sub> H <sub>4</sub> NHNHCSNHP) <sub>2</sub> ,                                | m. 180–2°. <sup>610</sup>   |
| C <sub>10</sub> H <sub>8</sub> (NHNHCSNHP) <sub>2</sub> -2,3,                            | m. 166° dec. <sup>562</sup> |
| <i>p,p'</i> -CH <sub>2</sub> (C <sub>6</sub> H <sub>4</sub> NMeNHNHCSNHP) <sub>2</sub> , | m. 211°. <sup>526</sup>     |

## MELTING POINTS OF SOME S-DERIVATIVES

S-Methyl, HI, m. 140°. <sup>83c</sup>

S-Methyl, 1-Benzoyl, m. 233° dec. <sup>230b</sup>

1-(*p*-Anisoyl), m. 244° dec. <sup>230b</sup>

1-(*p*-Nitrobenzoyl), m. 176° dec. <sup>230b</sup>

1-(*p*-Aminobenzenesulfonyl), m. 150–3°. <sup>194b</sup>

2-Methyl, H<sub>2</sub>SO<sub>4</sub>, m. 138–40° dec. <sup>194b</sup>

4-Methyl, HI, m. 160–3° dec. <sup>194b</sup>

1-Benzoyl, 4-Methyl, m. 140° dec. <sup>230b</sup>

1-Benzoyl, 4-(*i*-Propyl), m. 145° dec. <sup>230b</sup>

1-Benzoyl, 4-Phenyl, m. 205–7°. <sup>230b</sup>

1-Benzoyl, 4-Benzoyl, m. 171°. <sup>230b</sup>

1-(*p*-Anisoyl), 4-Methyl, m. 166° dec. <sup>230b</sup>

1-(*p*-Acetaminobenzenesulfonyl), 4-Methyl, HI, m. 208–10° dec. <sup>194b</sup>

1-(*p*-Aminobenzenesulfonyl), m. 150–2°. <sup>194b</sup>

S-Phenyl, 1-Phenyl, 4-Phenyl, m. 75°. <sup>68</sup>

**Physical Properties of Thiosemicarbazones**

A study was made of the phototropic and thermochromic properties of a number of thiosemicarbazones. <sup>181</sup> The absorption spectra have been useful for estimation and determination of photolability. <sup>49, 436b</sup> Absorption curves for thiosemicarbazones between 4500–2200 Å for a number of aldehydes have been observed. <sup>193</sup> The ultraviolet <sup>116, 130, 186, 478</sup> and infrared <sup>48, 130</sup> spectra have been determined for a number of the compounds. The thiosemicarbazones of saturated aldehydes and ketones have absorption bands near 2700 Å, which are 500 times as intense as those of the semicarbazones. <sup>128</sup>

## THIOSEMICARBAZONES OF ALDEHYDES

1. Acetaldehyde, m. 146°. <sup>160c</sup>

2. Propionaldehyde, m. 155°. <sup>193</sup>

3. Valeraldehyde, m. 65°. <sup>346, 347</sup>

4. *i*-Valeraldehyde, m. 53°. <sup>346, 347</sup>

5. Heptaldehyde, m. 79°. <sup>87</sup>

6. Pentadecanal, m. 96.5°. <sup>584</sup>

7. Palmitaldehyde, m. 109°. <sup>142</sup>

8. Stearaldehyde, m.  $111^{\circ}$ .<sup>142</sup>
9. Crotonaldehyde, m.  $167^{\circ}$ ,<sup>193</sup>  $160^{\circ}$ ; dimer, m.  $176^{\circ}$ ; trimer, m.  $222^{\circ}$ .<sup>281</sup>
10. Hexadienal, m.  $153^{\circ}$ .<sup>281</sup>
11. Octatrienal, m.  $156-8^{\circ}$ .<sup>281</sup>
12. 6-Diethylaminotetrahydrobenzaldehyde, m.  $106-8^{\circ}$ .<sup>281</sup>
13. 6-Diethylaminotetrahydro-*o*-tolualdehyde, m.  $149-52^{\circ}$ .<sup>281</sup>
14. Dihydrobenzaldehyde, m.  $102-4^{\circ}$ .<sup>281</sup>
15. Dihydro-*o*-tolualdehyde, m.  $147^{\circ}$ .<sup>281</sup>
16.  $\overline{\text{CMe}_2\text{CH}_2\text{CH}\cdot\text{CH}_2\text{CMe}\cdot\text{CCH}\cdot\text{CH}:\text{CHCHO}}$ , m.  $200^{\circ}$ .<sup>49</sup>
17. Citral, m.  $108^{\circ}$ ,<sup>87, 160c</sup>  $80-5^{\circ}$ .<sup>408</sup>
18.  $\beta$ -Cyclocitral, m.  $201^{\circ}$ .<sup>598</sup>
19. *D*-Citronellal, m.  $55^{\circ}$ .<sup>347</sup>
20. Glucose, m.  $167^{\circ}$ ; <sup>250</sup> *D*-, m.  $204^{\circ}$ ,<sup>347</sup>  $190^{\circ}$  dec.<sup>177b</sup>
21. Mannose, *D*-, m.  $187^{\circ}$ ,<sup>347</sup>  $176^{\circ}$  dec.; *L*-, m.  $174^{\circ}$  dec.<sup>177b</sup>
22. *D*-Galactose, m.  $148^{\circ}$ .<sup>347</sup>
23. Arabinose, *D*-, m.  $152^{\circ}$ ; *L*-, m.  $152^{\circ}$ ,<sup>177b</sup>  $148-50^{\circ}$ .<sup>235</sup>
24. Streptomycin, 3HCl, m.  $205^{\circ}$ .<sup>177b</sup>
25.  $\text{OCH}\cdot\text{CHO}$ , dec.  $300^{\circ}$ .<sup>347</sup>
26.  $\text{MeCO}\cdot\text{CHO}$ , m.  $257^{\circ}$ .<sup>599</sup>
27.  $\text{PhCOCHO}$ , m.  $170^{\circ}$ ,<sup>631</sup>  $156-8^{\circ}$ .<sup>235</sup>
28. Benzaldehyde, m.  $160^{\circ}$ ,<sup>49, 160c, 408, 633</sup>  $163^{\circ}$ .<sup>235</sup>
29. *o*- $\text{HOC}_6\text{H}_4\text{CHO}$ , m.  $235^{\circ}$ ,<sup>408</sup>  $155^{\circ}$ .<sup>250</sup>
30. *m*- $\text{HOC}_6\text{H}_4\text{CHO}$ , m.  $174^{\circ}$ ,<sup>291</sup>  $158^{\circ}$ ,<sup>250</sup>  $137^{\circ}$ .<sup>406</sup>
31. *p*- $\text{HOC}_6\text{H}_4\text{CHO}$ , m.  $237^{\circ}$ .<sup>406</sup>
32. *p*- $\text{AcOC}_6\text{H}_4\text{CHO}$ , m. about  $213^{\circ}$ .<sup>138a</sup>
33. *p*-Nicotinoyloxy  $\text{C}_6\text{H}_4\text{CHO}$ , m.  $228^{\circ}$ .<sup>596</sup>
34. *p*-Isonicotinoyloxy  $\text{C}_6\text{H}_4\text{CHO}$ , m.  $213^{\circ}$ .<sup>596</sup>
35. 2,4- $(\text{HO})_2\text{C}_6\text{H}_3\text{CHO}$ , m.  $240^{\circ}$ ,<sup>406</sup>  $235^{\circ}$ ,<sup>30</sup>  $231^{\circ}$  dec.<sup>291</sup>
36. 2,5- $(\text{HO})_2\text{C}_6\text{H}_3\text{CHO}$ , m.  $24^{\circ}$  dec.<sup>291</sup>
37. *o*- $\text{MeOC}_6\text{H}_4\text{CHO}$ , m.  $220^{\circ}$ ,<sup>406</sup>  $144^{\circ}$ .<sup>250</sup>
38. *m*- $\text{MeOC}_6\text{H}_4\text{CHO}$ , m.  $192^{\circ}$ .<sup>291</sup>
39. *p*- $\text{MeOC}_6\text{H}_4\text{CHO}$ , m.  $188^{\circ}$ ,<sup>390</sup>  $178^{\circ}$ ,<sup>406</sup>  $84^{\circ}$ .<sup>250</sup>
40. *o*- $\text{EtOC}_6\text{H}_4\text{CHO}$ , m.  $181^{\circ}$ .<sup>406</sup>
41. *p*- $\text{EtOC}_6\text{H}_4\text{CHO}$ , m.  $157^{\circ}$ ,<sup>138a</sup>  $152^{\circ}$ .<sup>291</sup>
42. *p*- $\text{BuOC}_6\text{H}_4\text{CHO}$ , m. about  $172^{\circ}$ .<sup>138a</sup>
43. *p*-(*i*-BuO) $\text{C}_6\text{H}_4\text{CHO}$ , m.  $148.5^{\circ}$ .<sup>37b</sup>
44. *p*-(*s*-BuO) $\text{C}_6\text{H}_4\text{CHO}$ , m.  $112.5^{\circ}$ .<sup>37b</sup>
45. *p*-(*i*-AmO) $\text{C}_6\text{H}_4\text{CHO}$ , m.  $135^{\circ}$ .<sup>37b</sup>
46. *p*-HexOC $_6\text{H}_4\text{CHO}$ , m.  $126^{\circ}$ .<sup>368</sup>

47. *p*-OctOC<sub>6</sub>H<sub>4</sub>CHO, m. about 110°. <sup>138a</sup>
48. *p*-C<sub>12</sub>H<sub>25</sub>OC<sub>6</sub>H<sub>4</sub>CHO, m. 114°. <sup>70</sup>
49. *p*-C<sub>18</sub>H<sub>37</sub>OC<sub>6</sub>H<sub>4</sub>CHO, m. 118°. <sup>70</sup>
50. *p*-[*c*-Pentyl(CH<sub>2</sub>)<sub>13</sub>O]C<sub>6</sub>H<sub>4</sub>CHO, m. 106°. <sup>70</sup>
51. *m*-AllylOC<sub>6</sub>H<sub>4</sub>CHO, m. 147°. <sup>131c, 413c</sup>
52. *p*-AllylOC<sub>6</sub>H<sub>4</sub>CHO, m. 160°, <sup>131c, 413c</sup> about 160°. <sup>138a</sup>
53. *m*-CrotylOC<sub>6</sub>H<sub>4</sub>CHO, m. 152°. <sup>131c, 413c</sup>
54. *p*-CrotylOC<sub>6</sub>H<sub>4</sub>CHO, m. 195°. <sup>131c, 413c</sup>
55. *m*-PropargylOC<sub>6</sub>H<sub>4</sub>CHO, m. 191°. <sup>131c</sup>
56. *p*-PropargylOC<sub>6</sub>H<sub>4</sub>CHO, m. 191°. <sup>413c</sup>
57. *p*-(2,3,4,6-Tetraacetyl-β-D-glucosidoxy)C<sub>6</sub>H<sub>4</sub>CHO, m. 212°. <sup>256</sup>
58. *p*-(1-Thioureido-β-D-glucosyl)C<sub>6</sub>H<sub>4</sub>CHO, m. 175°. <sup>256</sup>
59. *p*-(2,3,4,6)-Tetraacetyl-1-thioureido-β-D-glucosidoxy)C<sub>6</sub>H<sub>4</sub>-CHO, m. 165°. <sup>256</sup>
60. *p*-PhOC<sub>6</sub>H<sub>4</sub>CHO, m. 142°. <sup>131e</sup>
61. *p*-(*p*-MeOC<sub>6</sub>H<sub>4</sub>O)C<sub>6</sub>H<sub>4</sub>CHO, m. 174°. <sup>131e</sup>
62. *p*-(*p*-ClC<sub>6</sub>H<sub>4</sub>O)C<sub>6</sub>H<sub>4</sub>CHO, m. 172°. <sup>131e</sup>
63. *p*-(*o*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>O)C<sub>6</sub>H<sub>4</sub>CHO, m. 201°. <sup>131e</sup>
64. *m*-[2,4-(O<sub>2</sub>N)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>O]C<sub>6</sub>H<sub>4</sub>CHO, m. 228°. <sup>435</sup>
65. *p*-(*p*-H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>O)C<sub>6</sub>H<sub>4</sub>CHO, m. 203° dec. <sup>435</sup>
66. *p*-(*p*-AcNHC<sub>6</sub>H<sub>4</sub>O)C<sub>6</sub>H<sub>4</sub>CHO, m. 220° dec. <sup>435</sup>
67. *p*-(3,5-Me<sub>2</sub>C<sub>6</sub>H<sub>3</sub>O)C<sub>6</sub>H<sub>4</sub>CHO, m. 166°. <sup>435</sup>
68. *p*-PhCH<sub>2</sub>OC<sub>6</sub>H<sub>4</sub>CHO, m. 187°, <sup>435</sup> about 189°, <sup>138a</sup> 188°. <sup>70</sup>
69. *p*-(*p*-MeOC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>O)C<sub>6</sub>H<sub>4</sub>CHO, m. 211°, <sup>138a</sup> 170°. <sup>70</sup>
70. *p*-(*o*-ClC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>O)C<sub>6</sub>H<sub>4</sub>CHO, m. 165°. <sup>138a</sup>
71. *p*-(*m*-ClC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>O)C<sub>6</sub>H<sub>4</sub>CHO, m. 145°. <sup>138a</sup>
72. *p*-(*p*-ClC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>O)C<sub>6</sub>H<sub>4</sub>CHO, m. 189°, <sup>138a</sup> 195°, <sup>435</sup> 194°. <sup>70</sup>
73. *p*-(*p*-BrC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>O)C<sub>6</sub>H<sub>4</sub>CHO, m. 187°. <sup>435, 70</sup>
74. *p*-(*o*-IC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>O)C<sub>6</sub>H<sub>4</sub>CHO, m. 185°. <sup>138a</sup>
75. *p*-(*m*-IC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>O)C<sub>6</sub>H<sub>4</sub>CHO, m. 177°. <sup>138a</sup>
76. *p*-(*p*-IC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>O)C<sub>6</sub>H<sub>4</sub>CHO, m. 207°. <sup>138a</sup>
77. *p*-(*o*-MeC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>O)C<sub>6</sub>H<sub>4</sub>CHO, m. 188°. <sup>138a</sup>
78. *p*-(*m*-MeC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>O)C<sub>6</sub>H<sub>4</sub>CHO, m. 139°. <sup>138a</sup>
79. *p*-(*p*-MeC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>O)C<sub>6</sub>H<sub>4</sub>CHO, m. 191°, <sup>138a</sup> 185°. <sup>435</sup>
80. *p*-(*p*-EtC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>O)C<sub>6</sub>H<sub>4</sub>CHO, m. 168°, <sup>435</sup> 169°. <sup>70</sup>
81. *p*-(2,4-Me<sub>2</sub>C<sub>6</sub>H<sub>3</sub>CH<sub>2</sub>O)C<sub>6</sub>H<sub>4</sub>CHO, m. 152°, <sup>435</sup> 154°. <sup>70</sup>
82. *p*-(3,5-Me<sub>2</sub>C<sub>6</sub>H<sub>3</sub>CH<sub>2</sub>O)C<sub>6</sub>H<sub>4</sub>CHO, m. 183°. <sup>138a</sup>
83. *p*-PhCH<sub>2</sub>CH<sub>2</sub>OC<sub>6</sub>H<sub>4</sub>CHO, m. 172°. <sup>70</sup>
84. *p*-PhCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>OC<sub>6</sub>H<sub>4</sub>CHO, m. 144°. <sup>70</sup>



85.  $p$ -PhCH:CHCH<sub>2</sub>OC<sub>6</sub>H<sub>4</sub>CHO, m. 203°. <sup>70</sup>
86.  $p$ -( $\alpha$ -C<sub>10</sub>H<sub>7</sub>CH<sub>2</sub>O)C<sub>6</sub>H<sub>4</sub>CHO, m. 171°, <sup>435</sup> 172°. <sup>70</sup>
87.  $p$ -( $\alpha$ -C<sub>10</sub>H<sub>7</sub>CH<sub>2</sub>CH<sub>2</sub>O)C<sub>6</sub>H<sub>4</sub>CHO, m. 172°, <sup>435</sup> 173°. <sup>70</sup>
88. 2,3-(MeO)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>CHO, m. 231°. <sup>406</sup>
89. 2,4-(MeO)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>CHO, m. 198° dec. <sup>291</sup>
90. 3,4-(MeO)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>CHO, m. 207°. <sup>406</sup>
91. 3,5-(MeO)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>CHO, m. 212°. <sup>192</sup>
92. 3,4-(EtO)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>CHO, m. 176°. <sup>406</sup>
93. 3,4-(MeO)(PhCH<sub>2</sub>O)C<sub>6</sub>H<sub>3</sub>CHO, m. 165°. <sup>435</sup>
94. 5,4-(MeO)(PhCH<sub>2</sub>O)C<sub>6</sub>H<sub>3</sub>CHO, m. 166°. <sup>70</sup>
95. 3,4-CH<sub>2</sub>O<sub>2</sub>C<sub>6</sub>H<sub>3</sub>CHO (piperonal), m. 191°, <sup>406</sup> 185°, <sup>347</sup> 183°. <sup>87</sup>
96. 3,4,5-(MeO)<sub>3</sub>C<sub>6</sub>H<sub>2</sub>CHO, m. 216°. <sup>291</sup>
97.  $p$ -(Dihydrochaulmoogrylo)C<sub>6</sub>H<sub>4</sub>CHO, m. 105°. <sup>435</sup>
98.  $p$ -MeSC<sub>6</sub>H<sub>4</sub>CHO, m. 177-9°. <sup>536</sup>
99.  $p$ -MeSO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>CHO, m. 232° dec. <sup>614</sup>
100.  $p$ -EtSC<sub>6</sub>H<sub>4</sub>CHO, m. 145°. <sup>174</sup>
101.  $p$ -EtSOC<sub>6</sub>H<sub>4</sub>CHO, dec. 210°. <sup>174</sup>
102.  $p$ -EtSO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>CHO, m. 234°, <sup>291</sup> 224°, <sup>614</sup> dec. 229-30°. <sup>174</sup>
103.  $p$ -PrSC<sub>6</sub>H<sub>4</sub>CHO, m. 121°. <sup>174</sup>
104.  $p$ -PrSOC<sub>6</sub>H<sub>4</sub>CHO, m. 171°. <sup>174</sup>
105.  $p$ -PrSO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>CHO, m. 208°. <sup>174</sup>
106.  $p$ -Me<sub>2</sub>CHSC<sub>6</sub>H<sub>4</sub>CHO, m. 138°. <sup>174</sup>
107.  $p$ -Me<sub>2</sub>CHSOC<sub>6</sub>H<sub>4</sub>CHO, dec. 190.5°. <sup>174</sup>
108.  $p$ -Me<sub>2</sub>CHSO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>CHO, m. 218.5°. <sup>174</sup>
109.  $p$ -BuSC<sub>6</sub>H<sub>4</sub>CHO, m. 118°. <sup>174</sup>
110.  $p$ -BuSOC<sub>6</sub>H<sub>4</sub>CHO, m. 171°. <sup>174</sup>
111.  $p$ -BuSO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>CHO, m. 170°, <sup>174</sup> 192°, <sup>291</sup> 185°. <sup>614</sup>
112.  $p$ -Me<sub>2</sub>CHCH<sub>2</sub>SC<sub>6</sub>H<sub>4</sub>CHO, m. 145°. <sup>174</sup>
113.  $p$ -Me<sub>2</sub>CHCH<sub>2</sub>SOC<sub>6</sub>H<sub>4</sub>CHO, m. 179-81°. <sup>174</sup>
114.  $p$ -Me<sub>2</sub>CHCH<sub>2</sub>SO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>CHO, m. 179°. <sup>174</sup>
115.  $p$ -HexSO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>CHO, m. 182-4°. <sup>614</sup>
116.  $p$ -( $p$ -O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>SO<sub>2</sub>)C<sub>6</sub>H<sub>4</sub>CHO, m. 245° dec. <sup>611</sup>
117.  $p$ -( $p$ -AcNHC<sub>6</sub>H<sub>4</sub>S)C<sub>6</sub>H<sub>4</sub>CHO, m. 216°. <sup>611</sup>
118.  $p$ -( $p$ -AcNHC<sub>6</sub>H<sub>4</sub>SO<sub>2</sub>)C<sub>6</sub>H<sub>4</sub>CHO, m. 195°. <sup>611</sup>
119. 3,4-Me(MeS)C<sub>6</sub>H<sub>3</sub>CHO, m. 208° dec. <sup>536</sup>
120.  $p$ -HOOCCH<sub>2</sub>SO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>CHO, m. 229°. <sup>614</sup>
121.  $p$ -MeC<sub>6</sub>H<sub>4</sub>SO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>CHO, mono, m. 270°; di, m. 259° dec. <sup>577</sup>
122. Ph<sub>3</sub>SiC<sub>6</sub>H<sub>4</sub>CHO, m. 235°. <sup>525</sup>
123.  $o$ -ClC<sub>6</sub>H<sub>4</sub>CHO, m. 223°, <sup>406</sup> 210°. <sup>329</sup>

124.  $p\text{-ClC}_6\text{H}_4\text{CHO}$ , m.  $220^\circ$ .<sup>406</sup>
125.  $2,4\text{-Cl}_2\text{C}_6\text{H}_3\text{CHO}$ , m.  $246^\circ$ .<sup>406</sup>
126.  $3,4\text{-Cl}_2\text{C}_6\text{H}_3\text{CHO}$ , m.  $181^\circ$ .<sup>406</sup>
127.  $p\text{-F}_3\text{CC}_6\text{H}_4\text{CHO}$ , m.  $173^\circ$ .<sup>528</sup>
128.  $o\text{-O}_2\text{NC}_6\text{H}_4\text{CHO}$ , m.  $267^\circ$ ,<sup>193</sup>  $266^\circ$ ,<sup>406</sup>  $242^\circ$ ,<sup>329</sup>  $195^\circ$ .<sup>390</sup>
129.  $m\text{-O}_2\text{NC}_6\text{H}_4\text{CHO}$ , m.  $240^\circ$ ,<sup>406</sup>  $239^\circ$ ,<sup>193</sup>  $209^\circ$ .<sup>390</sup>
130.  $p\text{-O}_2\text{NC}_6\text{H}_4\text{CHO}$ , m.  $262^\circ$ ,<sup>193</sup>  $255^\circ$ ,<sup>406</sup>  $231^\circ$ ,<sup>87</sup>  $225\text{--}7^\circ$ ,<sup>506</sup>  
 $207^\circ$ ,<sup>390</sup>  $109^\circ$ .<sup>250</sup>
131.  $p\text{-H}_2\text{NC}_6\text{H}_4\text{CHO}$ , m.  $204^\circ$ ,<sup>32b, 406</sup>  $198^\circ$ .<sup>349c</sup>
132.  $p\text{-AcNHC}_6\text{H}_4\text{CHO}$ , m.  $247^\circ$ ,<sup>406</sup>  $234^\circ$  dec.,<sup>506</sup>  $227^\circ$  dec.,<sup>632</sup>  
 $220\text{--}4^\circ$ ,<sup>1</sup>  $207^\circ$ ,<sup>87</sup>  $170^\circ$ .<sup>250</sup>
133. -SAg salt of 132, m.  $252^\circ$  dec.<sup>220</sup>
134.  $p\text{-AmCONHC}_6\text{H}_4\text{CHO}$ , m.  $234^\circ$  dec.<sup>349b</sup>
135.  $p\text{-MeCH:CHCONHC}_6\text{H}_4\text{CHO}$ , m.  $240^\circ$  dec.<sup>349c</sup>
136.  $p\text{-CH}_2\text{:CMeCONHC}_6\text{H}_4\text{CHO}$ , m.  $216^\circ$  dec.<sup>349c</sup>
137.  $p\text{-Me}_2\text{C:CHCONHC}_6\text{H}_4\text{CHO}$ , m.  $224^\circ$  dec.<sup>349b</sup>
138.  $p\text{-AcCH}_2\text{CONHC}_6\text{H}_4\text{CHO}$ , m.  $190^\circ$ .<sup>32b</sup>
139.  $p\text{-HOOCCH}_2\text{CH}_2\text{CONHC}_6\text{H}_4\text{CHO}$ , m.  $176^\circ$  dec.<sup>250</sup>
140.  $p\text{-(}o\text{-HOOC}_6\text{H}_4\text{CONH)C}_6\text{H}_4\text{CHO}$ , m.  $225^\circ$ .<sup>242</sup>
141.  $p\text{-NicotinoylaminoC}_6\text{H}_4\text{CHO}$ , m.  $260\text{--}2^\circ$  dec.<sup>596</sup>
142.  $p\text{-IsonicotinoylaminoC}_6\text{H}_4\text{CHO}$ , m.  $280^\circ$ .<sup>596</sup>
143. Taurylamido $\text{C}_6\text{H}_4\text{CHO}$ , m.  $188.3^\circ$  dec.; HCl, m.  $228^\circ$ ; pic-  
rate, m.  $223^\circ$ .<sup>453</sup>
144.  $p\text{-AcNHC}_6\text{H}_4\text{SO}_2\text{NHC}_6\text{H}_4\text{CHO}$ , m.  $240^\circ$ .<sup>390</sup>
145.  $p\text{-MeAcNC}_6\text{H}_4\text{CHO}$ , m.  $214\text{--}16^\circ$ .<sup>133a</sup>
146.  $p\text{-EtAcNC}_6\text{H}_4\text{CHO}$ , m.  $216^\circ$ .<sup>133a</sup>
147.  $p\text{-PrAcNC}_6\text{H}_4\text{CHO}$ , m.  $208\text{--}10^\circ$ .<sup>133a</sup>
148.  $p\text{-Me}_2\text{CHAcNC}_6\text{H}_4\text{CHO}$ , m.  $210\text{--}12^\circ$ .<sup>133a</sup>
149.  $p\text{-BuNHC}_6\text{H}_4\text{CHO}$ , m.  $208^\circ$ .<sup>616</sup>
150.  $p\text{-BuAcNC}_6\text{H}_4\text{CHO}$ , m.  $201\text{--}3^\circ$ .<sup>133a</sup>
151.  $p\text{-C}_8\text{H}_{17}\text{NHC}_6\text{H}_4\text{CHO}$ , m.  $166^\circ$ .<sup>616</sup>
152.  $p\text{-AllylAcNC}_6\text{H}_4\text{CHO}$ , m.  $211\text{--}13^\circ$ .<sup>133a</sup>
153.  $p\text{-MorpholinoC}_6\text{H}_4\text{CHO}$ , m.  $230^\circ$ .<sup>616</sup>
154.  $p\text{-(2-Pyridylamino)C}_6\text{H}_4\text{CHO}$ , m.  $217^\circ$ .<sup>616</sup>
155.  $p\text{-(}p\text{-MeOC}_6\text{H}_4\text{NH)C}_6\text{H}_4\text{CHO}$ , dec.  $212^\circ$ .<sup>616</sup>
156.  $p\text{-(}p\text{-O}_2\text{NC}_6\text{H}_4\text{NH)C}_6\text{H}_4\text{CHO}$ , dec.  $213\text{--}15^\circ$ .<sup>616</sup>
157.  $p\text{-[3,4-HO(HOOC)C}_6\text{H}_3\text{NH]C}_6\text{H}_4\text{CHO}$ , dec.  $233^\circ$ .<sup>616</sup>
158.  $p\text{-Me}_2\text{NC}_6\text{H}_4\text{CHO}$ , m.  $217^\circ$ ,<sup>242</sup>  $211^\circ$ ,<sup>390</sup>  $209^\circ$ ,<sup>542</sup>  $208^\circ$ ,<sup>406</sup>  
 $125^\circ$ .<sup>250</sup>
159.  $p\text{-Et}_2\text{NC}_6\text{H}_4\text{CHO}$ , m.  $193^\circ$ ,<sup>390</sup>  $180^\circ$ ,<sup>506</sup>  $178^\circ$ ,<sup>406</sup>  $206\text{--}8^\circ$  dec.<sup>616</sup>

160. *p*-Pr<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO, m. 196°. <sup>616</sup>
161. *p*-(C<sub>8</sub>H<sub>17</sub>)<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO, m. 170°. <sup>616</sup>
162. *p*-(HOCH<sub>2</sub>CH<sub>2</sub>)<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO, m. 203°. <sup>616</sup>
163. 3,4-H<sub>2</sub>N(AcNH)C<sub>6</sub>H<sub>3</sub>CHO, m. 320° dec. <sup>613</sup>
164. 3,4-(AcNH)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>CHO, m. 276°. <sup>613</sup>
165. *p*-H<sub>2</sub>NNHC<sub>6</sub>H<sub>4</sub>CHO, m. 102°. <sup>455a</sup>
166. *p*-MeNHCONHC<sub>6</sub>H<sub>4</sub>CHO, m. 220° dec. <sup>624</sup>
167. *p*-PrNHCONHC<sub>6</sub>H<sub>4</sub>CHO, m. 205° dec. <sup>624</sup>
168. *p*-Me<sub>2</sub>CHNHCONHC<sub>6</sub>H<sub>4</sub>CHO, m. 198° dec. <sup>624</sup>
169. *p*-BuNHCONHC<sub>6</sub>H<sub>4</sub>CHO, m. 195° dec. <sup>624</sup>
170. *p*-Me<sub>2</sub>CHCH<sub>2</sub>NHCONHC<sub>6</sub>H<sub>4</sub>CHO, m. 206° dec. <sup>624</sup>
171. *p*-PhNHCONHC<sub>6</sub>H<sub>4</sub>CHO, m. 245° dec. <sup>624</sup>
172. *p*-(2-Pyridyl)C<sub>6</sub>H<sub>4</sub>CHO, m. 215°. <sup>102</sup>
173. *p*-NaO<sub>3</sub>SC<sub>6</sub>H<sub>4</sub>CHO, dec. 230°. <sup>616</sup>
174. *p*-H<sub>2</sub>NSO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>CHO, m. 235°, <sup>299a</sup> 231° dec., <sup>425</sup> 227°. <sup>616</sup>
175. *p*-PhNHSO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>CHO, m. 207° dec. <sup>425</sup>
176. *p*-(*p*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>NHSO<sub>2</sub>)C<sub>6</sub>H<sub>4</sub>CHO, m. 236° dec. <sup>425</sup>
177. *p*-(*p*-EtOC<sub>6</sub>H<sub>4</sub>NHSO<sub>2</sub>)C<sub>6</sub>H<sub>4</sub>CHO, m. 209° dec. <sup>425</sup>
178. *p*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>COC<sub>6</sub>H<sub>4</sub>CHO, m. 233° dec. <sup>611</sup>
179. *m*-C<sub>6</sub>H<sub>4</sub>(CHO)<sub>2</sub>, di-thiosemicarbazone, m. 247° dec. <sup>413a</sup>
180. *p*-C<sub>6</sub>H<sub>4</sub>(CHO)<sub>2</sub>, mono-thiosemicarbazone, m. 215° dec. <sup>413a</sup>
181. *p*-H<sub>2</sub>NN:CHC<sub>6</sub>H<sub>4</sub>CHO, m. 280°. <sup>131h</sup>
182. *p*-PhNHN:CHC<sub>6</sub>H<sub>4</sub>CHO, m. 234°. <sup>131h</sup>
183. *p,p'*-(N:CHC<sub>6</sub>H<sub>4</sub>CHO), di-thiosemicarbazone, m. 280°. <sup>131h</sup>

#### SUBSTITUTED HYDROXYBENZALDEHYDES

184. 2,3-HO(MeO)C<sub>6</sub>H<sub>3</sub>CHO, m. 246°. <sup>406</sup>
185. 2,4-HO(MeO)C<sub>6</sub>H<sub>3</sub>CHO, m. 214° dec. <sup>521</sup>
186. 4,2-HO(MeO)C<sub>6</sub>H<sub>3</sub>CHO, m. 196.5°. <sup>521</sup>
187. 4,3-HO(MeO)C<sub>6</sub>H<sub>3</sub>CHO (vanillin), m. 200°, <sup>406</sup> 197°, <sup>160c</sup> 194°, <sup>87</sup> 190-4°. <sup>299a</sup>
188. 4,3-HO(EtO)C<sub>6</sub>H<sub>3</sub>CHO, m. 203°. <sup>406</sup>
189. 5,2-Cl(HO)C<sub>6</sub>H<sub>3</sub>CHO, m. 282°. <sup>406</sup>
190. 3,4-Cl(HO)C<sub>6</sub>H<sub>3</sub>CHO, m. 211°, <sup>435</sup> 237°. <sup>70</sup>
191. 3,5,4-Cl<sub>2</sub>(HO)C<sub>6</sub>H<sub>2</sub>CHO, m. 260°. <sup>70</sup>
192. 5,2-Br(HO)C<sub>6</sub>H<sub>3</sub>CHO, m. 224°, <sup>390</sup> 218-20°. <sup>497</sup>
193. 3,4-Br(HO)C<sub>6</sub>H<sub>3</sub>CHO, m. 220°. <sup>435</sup>
194. 3,5,4-Br<sub>2</sub>(HO)C<sub>6</sub>H<sub>2</sub>CHO, dec. 235°, <sup>435</sup> m. 256° dec. <sup>70</sup>
195. 3,5,2-I<sub>2</sub>(HO)C<sub>6</sub>H<sub>2</sub>CHO, m. 230°, <sup>390</sup> 225°. <sup>87</sup>
196. 3,5,4-I<sub>2</sub>(HO)C<sub>6</sub>H<sub>2</sub>CHO, dec. 230°, <sup>435</sup> m. 266°. <sup>70</sup>

197. 5,2,3-Br(HO)(MeO)C<sub>6</sub>H<sub>2</sub>CHO, m. 210–2°.<sup>497</sup> (5 bromo-vanillin)  
198. 5,4,3-Br(HO)(MeO)C<sub>6</sub>H<sub>2</sub>CHO, m. 224–6°.<sup>497</sup>  
199. 4,2-O<sub>2</sub>N(HO)C<sub>6</sub>H<sub>3</sub>CHO, m. 230° dec.,<sup>30</sup> 243° dec.<sup>621</sup>  
200. 4,2-O<sub>2</sub>N(AcO)C<sub>6</sub>H<sub>3</sub>CH(OAc)<sub>2</sub>, m. 230° dec.<sup>571</sup>

## SUBSTITUTED ETHER-BENZALDEHYDES

201. 3,4-Cl(PhCH<sub>2</sub>O)C<sub>6</sub>H<sub>3</sub>CHO, m. 179°,<sup>435</sup> 180°.<sup>70</sup>  
202. 3,4-Br(PhCH<sub>2</sub>O)C<sub>6</sub>H<sub>3</sub>CHO, m. 178°,<sup>435</sup> 183°.<sup>70</sup>  
203. 3,5,4-Br<sub>2</sub>(PhCH<sub>2</sub>O)C<sub>6</sub>H<sub>3</sub>CHO, m. 218°.<sup>70</sup>  
204. 3,5,4-I<sub>2</sub>(PhCH<sub>2</sub>O)C<sub>6</sub>H<sub>2</sub>CHO, dec. 160°,<sup>435</sup> m. 214°.<sup>70</sup>  
205. 5,2,3-Br(MeO)<sub>2</sub>C<sub>6</sub>H<sub>2</sub>CHO, m. 220–2°.<sup>497</sup>  
206. 5,3,4-Br(MeO)<sub>2</sub>C<sub>6</sub>H<sub>2</sub>CHO, m. 181–3°.<sup>497</sup>  
207. 5,3,4-Br(CH<sub>2</sub>O<sub>2</sub>)C<sub>6</sub>H<sub>2</sub>CHO, m. about 250°.<sup>497</sup>  
208. 4,2-O<sub>2</sub>N(MeO)C<sub>6</sub>H<sub>3</sub>CHO, m. 250°.<sup>260</sup>  
209. 4,2-O<sub>2</sub>N(EtO)C<sub>6</sub>H<sub>3</sub>CHO, m. 228–30°.<sup>260</sup>  
210. 4,2-O<sub>2</sub>N(PrO)C<sub>6</sub>H<sub>3</sub>CHO, m. 225°.<sup>260</sup>  
211. 4,2-O<sub>2</sub>N(BuO)C<sub>6</sub>H<sub>3</sub>CHO, m. 214.5°.<sup>260</sup>

## SUBSTITUTED AMINOBENZALDEHYDES

212. 4,2-H<sub>2</sub>N(HO)C<sub>6</sub>H<sub>3</sub>CHO, m. 217° dec.,<sup>30</sup> 216° dec.<sup>571</sup>  
213. 4,2-AcNH(HO)C<sub>6</sub>H<sub>3</sub>CHO, m. 252° dec.,<sup>30</sup> 260–4° dec.<sup>191</sup>  
214. 4,2-BzNH(HO)C<sub>6</sub>H<sub>3</sub>CHO, m. 180–6° dec.<sup>191</sup>  
215. 4,2-HexNH(HO)C<sub>6</sub>H<sub>3</sub>CHO, m. 260.6°.<sup>191</sup>  
216. 4,2-(*o*-HOC<sub>6</sub>H<sub>4</sub>NH)(HO)C<sub>6</sub>H<sub>3</sub>CHO, sinters 200–10° dec.<sup>191</sup>  
217. 4,2-Me<sub>2</sub>N(HO)C<sub>6</sub>H<sub>3</sub>CHO, m. 235° dec.<sup>30</sup>  
218. 4,2-H<sub>2</sub>N(MeO)C<sub>6</sub>H<sub>3</sub>CHO, m. 164°.<sup>613</sup>  
219. 4,2-AcNH(MeO)C<sub>6</sub>H<sub>3</sub>CHO, m. 233°,<sup>260</sup> 232° dec.,<sup>30</sup> 212°.<sup>613</sup>  
220. 4,2-Succinylamido(Me)C<sub>6</sub>H<sub>3</sub>CHO, m. 190°.<sup>613</sup>  
221. 4,3-H<sub>2</sub>N(MeO)C<sub>6</sub>H<sub>3</sub>CHO, m. 161°.<sup>613</sup>  
222. 4,3-AcNH(MeO)C<sub>6</sub>H<sub>3</sub>CHO, m. 233°.<sup>613</sup>  
223. 4,3-BzNH(MeO)C<sub>6</sub>H<sub>3</sub>CHO, dec. 250°.<sup>613</sup>  
224. 4,3-Succinylamido(MeO)C<sub>6</sub>H<sub>3</sub>CHO, m. 201°.<sup>613</sup>  
225. 4,2-AcNH(MeO)C<sub>6</sub>H<sub>3</sub>CHO, m. 233°,<sup>260</sup> 232° dec.,<sup>30</sup> 212°.<sup>613</sup>  
226. 4,2-AcNH(EtO)C<sub>6</sub>H<sub>3</sub>CHO, m. 229–32°.<sup>259, 260</sup>  
227. 4,2-AcNH(PrO)C<sub>6</sub>H<sub>3</sub>CHO, m. 229.5°.<sup>259, 260</sup>  
228. 4,2-AcNH(BuO)C<sub>6</sub>H<sub>3</sub>CHO, m. 216°,<sup>259, 618</sup> 214°.<sup>260</sup>  
229. 4,2-AcNH(AmO)C<sub>6</sub>H<sub>3</sub>CHO, m. 174.5°.<sup>259, 260</sup>  
230. 4,2-AcNH(PhCH<sub>2</sub>O)C<sub>6</sub>H<sub>3</sub>CHO, m. 201°.<sup>613</sup>  
231. 4,2-AcNH(MeSO<sub>2</sub>)C<sub>6</sub>H<sub>3</sub>CHO, m. 256–8° dec.<sup>443</sup>

232. 4,2-AcNH(EtSO<sub>2</sub>)C<sub>6</sub>H<sub>3</sub>CHO, m. 241°. <sup>443</sup>
233. 4,2-AcNH(PhCH<sub>2</sub>SO<sub>2</sub>)C<sub>6</sub>H<sub>3</sub>CHO, m. 255–7°. <sup>443</sup>
234. 2,4-Cl(AcNH)C<sub>6</sub>H<sub>3</sub>CHO, m. 200° dec. <sup>543</sup>
235. 2,4,5 H<sub>2</sub>N(HO)(HOOC)C<sub>6</sub>H<sub>2</sub>N:NC<sub>6</sub>H<sub>4</sub>CHO, m. 215°. <sup>578</sup>
236. *o*-MeC<sub>6</sub>H<sub>4</sub>CHO, m. 166°. <sup>406</sup>
237. *m*-MeC<sub>6</sub>H<sub>4</sub>CHO, m. 179°. <sup>406</sup>
238. *p*-MeC<sub>6</sub>H<sub>4</sub>CHO, m. 170°. <sup>406</sup>
239. Cyclopolic acid, m. 182°. <sup>523</sup>
240. *p*-Me<sub>2</sub>CHC<sub>6</sub>H<sub>4</sub>CHO, m. 147°. <sup>406</sup>
241. 2,4-Allyl(AllylO)C<sub>6</sub>H<sub>3</sub>CHO, m. 169°. <sup>131c</sup>
242. 3,4-Allyl(AllylO)C<sub>6</sub>H<sub>3</sub>CHO, m. 169°. <sup>413c</sup>
243. *p*-PhCH:CHC<sub>6</sub>H<sub>4</sub>CHO, m. 265°. <sup>544</sup>
244. PhCH<sub>2</sub>CHO, m. 148°. <sup>87</sup>
245. 5-Benzylsalicylaldehyde, m. 202°. <sup>69</sup>
246. PhCH<sub>2</sub>CH<sub>2</sub>CHO, m. 113–15°. <sup>406</sup>
247. PhCH:CHCHO, m. 136°, <sup>406</sup> 123°, <sup>160c</sup> 95°. <sup>87</sup>
248. *p*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CH:CHCHO, m. 228° dec., <sup>131f</sup> 224° dec. <sup>349a</sup>
249. *p*-H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CH:CHCHO, m. 207°, <sup>349a</sup> 206°, <sup>131f</sup> 199°. <sup>4</sup>
250. *p*-AcNHC<sub>6</sub>H<sub>4</sub>CH:CHCHO, m. 222°, <sup>131f</sup> 212°, <sup>4</sup> 207°. <sup>77a</sup>
251. *p*-AmCONHC<sub>6</sub>H<sub>4</sub>CH:CHCHO, m. 201° dec. <sup>349b</sup>
252. *p*-MeCH:CHCONHC<sub>6</sub>H<sub>4</sub>CH:CHCHO, m. 234° dec. <sup>349c</sup>
253. *p*-Me<sub>2</sub>C:CHCONHC<sub>6</sub>H<sub>4</sub>CH:CHCHO, m. 235° dec. <sup>349b</sup>
254. *p*-BzNHC<sub>6</sub>H<sub>4</sub>CH:CHCHO, m. 234° dec. <sup>249b</sup>
255. *p*-PhCH<sub>2</sub>CONHC<sub>6</sub>H<sub>4</sub>CH:CHCHO, m. 223° dec. <sup>349b</sup>
256. *p*-PhCH:CHCONHC<sub>6</sub>H<sub>4</sub>CH:CHCHO, m. 243° dec. <sup>349c</sup>
257. *p*-HOOCCH<sub>2</sub>CH<sub>2</sub>CONHC<sub>6</sub>H<sub>4</sub>CH:CHCHO, m. 208° dec. <sup>349c</sup>
258. *p*-MeC<sub>6</sub>H<sub>4</sub>SO<sub>2</sub>NHC<sub>6</sub>H<sub>4</sub>CH:CHCHO, m. 242° dec. <sup>349b</sup>
259. *p*-MeOOCCH<sub>2</sub>CH:CHCHO, m. 224° dec. <sup>131f</sup>
260. *p*-HOOCCH<sub>2</sub>OC<sub>6</sub>H<sub>4</sub>CH:CHCHO, m. 202°. <sup>131f</sup>
261. *p*-EtSO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>CH:CHCHO, m. 190°. <sup>131f</sup>
262. *p*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CH:CB<sub>2</sub>CHO, m. 214°. <sup>349d</sup>
263. PhCH:CAmCHO, m. 142°. <sup>406</sup>
264. *p*-PhC<sub>6</sub>H<sub>4</sub>CHO, m. 202.5°. <sup>102</sup>
265. *p*-MeOC<sub>6</sub>H<sub>4</sub>C<sub>6</sub>H<sub>4</sub>CHO, m. 213°. <sup>102</sup>
266. *p*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>C<sub>6</sub>H<sub>4</sub>CHO, m. 240° dec. <sup>102</sup>
267. *p*-EtOOCCH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>C<sub>6</sub>H<sub>4</sub>CHO, m. 235° dec. <sup>102</sup>
268. Diphenylene oxide-3-carboxaldehyde, m. 226°. <sup>131e</sup>
269. α-C<sub>10</sub>H<sub>7</sub>CHO, m. 126°. <sup>250</sup>
270. β-C<sub>10</sub>H<sub>7</sub>CHO, m. 132°. <sup>250</sup>
271. 2,1-HOC<sub>10</sub>H<sub>6</sub>CHO, m. 275°, <sup>406</sup> 280°. <sup>236</sup>

272. 2,5,1-(HO)<sub>2</sub>C<sub>10</sub>H<sub>5</sub>CHO, m. 275°. <sup>576</sup>  
 273. 2,1-EtOC<sub>10</sub>H<sub>6</sub>CHO, m. 175°. <sup>291</sup>  
 274. 2,5,1-(MeO)<sub>2</sub>C<sub>10</sub>H<sub>5</sub>CHO, m. 224°. <sup>576</sup>  
 275. 4,5,1-(MeO)<sub>2</sub>C<sub>10</sub>H<sub>5</sub>CHO, m. 259°. <sup>537</sup>  
 276. 8,4,5,1-Me(MeO)<sub>2</sub>C<sub>10</sub>H<sub>4</sub>CHO, m. 257°. <sup>537</sup>  
 277. 6,2,1-Br(MeO)<sub>2</sub>C<sub>10</sub>H<sub>5</sub>CHO, m. 240°. <sup>576</sup>  
 278. 2,1-MeSC<sub>10</sub>H<sub>6</sub>CHO, m. 171°. <sup>538</sup>  
 279. Anthraldehyde, m. 195°. <sup>542</sup>  
 280. Phenylglyoxylic acid, m. 174-6°. <sup>235</sup>

## THIOSEMICARBAZONES OF KETONES

281. Acetone, m. 185°, <sup>406</sup> 180°, <sup>1</sup> 179°, <sup>87, 160c, 331</sup> 121°. <sup>444a</sup>  
 282. Butanone, m. 104-6°. <sup>331</sup>  
 283. Heptanone-2, m. 73°, <sup>87</sup> 64-6°. <sup>406</sup>  
 284. Heptanone-4, m. 81°. <sup>331</sup>  
 285. Octanone-2, m. 67°. <sup>331</sup>  
 286. 2,6-Dimethylheptanone-4, m. 104°. <sup>87</sup>  
 287. Chloropropanone, m. 146°. <sup>41</sup>  
 288. Phenoxypropanone, m. 136-8°. <sup>406</sup>  
 289. 2-Hydroxybutanone-3, m. 161°. <sup>235</sup>  
 290. 1-Hydroxyoctanone-2, m. 148.5°. <sup>574</sup>  
 291. 2-Azido-2-methylbutanone-3, m. 107°. <sup>561</sup>  
 292. Carbethoxypropanone, m. 97°. <sup>406</sup>  
 293. 1-Carboxybutanone-3, m. 195°. <sup>406</sup>  
 294. 1-Carbethoxybutanone-3, m. 117°. <sup>406</sup>  
 295. C<sub>6</sub>H<sub>8</sub>AcCOOH, m. 164°. <sup>516</sup>  
 296. Vitamin-C, m. 158-60°. <sup>406</sup>  
 297. α-Ionone, m. 121°. <sup>545</sup>  
 298. β-Ionone, m. 158°. <sup>545</sup>  
 299. β-Irone, m. 181°. <sup>545</sup>  
 300. c-Pentanone, m. 155-7°, <sup>87</sup> 154°. <sup>406</sup>  
 301. c-Hexanone, m. 165-7°, <sup>406</sup> 164°, <sup>193</sup> 148°. <sup>87</sup>  
 302. 2-Methyl-c-hexanone, m. 151°. <sup>87</sup>  
 303. 3-Methyl-c-hexanone, m. 118°. <sup>406</sup>  
 304. 4-Methyl-c-hexanone, m. 107°. <sup>406</sup>  
 305. *trans*-2-Methyl-1-hydroxyacetylcyclohexanol, m. 210°. <sup>522</sup>  
 306. Ethyl 2,5-c-hexanedionecarboxylate, *mono*, m. 85°; *bis*, m. 173°. <sup>519</sup>  
 307. Dehydrohydoxycholic acid, m. 168-70°. <sup>364</sup>  
 308. Isophorone, m. 181°. <sup>406</sup>

309. Camphor, m.  $191^{\circ}$ .<sup>406</sup>
310. *D*-Camphorquinone, m.  $174^{\circ}$ .<sup>148</sup>
311. *L*-Menthone, m.  $157^{\circ}$ .<sup>347</sup>
312. Piperitone, m.  $165^{\circ}$ .<sup>557, 619</sup>
313. Carone, m.  $132^{\circ}$ .<sup>185</sup>
314. 1-Methyl-*c*-pentene-3-ol-2-one, 2 thiosemicarbazones, m.  $225-7^{\circ}$  and  $242-4^{\circ}$ .<sup>207</sup>
315. 1-Methyl-*c*-pentene-3-ol-4-one, 2 thiosemicarbazones, m.  $222^{\circ}$  and  $232^{\circ}$ .<sup>207</sup>
316. 2-Oxo-*c*-hexanol, m.  $179^{\circ}$ .<sup>235</sup>
317. 8-Hydroxy-*p*-menthadien-2-one, m.  $181.5^{\circ}$ .<sup>558</sup>
318.  $\alpha$ -Tetrahydrosantonin, m.  $234^{\circ}$ .<sup>547</sup>
319. 2-Oxo-*myo*-inositol, m.  $196^{\circ}$ .<sup>282</sup>
320. Testosterone, m.  $161^{\circ}$ .<sup>84</sup>
321. Dihydroandrosterone-17-thiosemicarbazone, m.  $228-30^{\circ}$ .<sup>84</sup>
322. Progesterone-3,7-dithiosemicarbazone, m.  $260-2^{\circ}$ .<sup>84</sup>
323. Acetophenone, m.  $116^{\circ}$ ,<sup>331</sup>  $108^{\circ}$ ; <sup>87, 347</sup> antiform, m.  $119^{\circ}$ ; synform, m.  $90.5^{\circ}$ .<sup>406</sup>
324. *o*-HOC<sub>6</sub>H<sub>4</sub>COMe, m.  $195^{\circ}$ ,<sup>406</sup>  $196^{\circ}$ .<sup>291</sup>
325. 2,4-(HO)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>COMe, m.  $212-15^{\circ}$ .<sup>406</sup>
326. *o*-MeOC<sub>6</sub>H<sub>4</sub>COMe, m.  $177^{\circ}$ .<sup>291</sup>
327. *p*-MeOC<sub>6</sub>H<sub>4</sub>COMe, m.  $178^{\circ}$ ,<sup>291</sup>  $175^{\circ}$ ,<sup>87</sup>  $184-95^{\circ}$ ,<sup>406</sup>  $150^{\circ}$ .<sup>351</sup>
328. *p*-EtOC<sub>6</sub>H<sub>4</sub>COMe, m.  $155^{\circ}$ .<sup>291</sup>
329. *p*-PhCH<sub>2</sub>OC<sub>6</sub>H<sub>4</sub>COMe, m.  $177^{\circ}$ .<sup>70</sup>
330. 3,4-(MeO)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>COMe, m.  $227^{\circ}$  dec.<sup>70</sup>
331. 3,4-(MeO)(PhCH<sub>2</sub>O)C<sub>6</sub>H<sub>3</sub>COMe, m.  $175^{\circ}$ ,  $88^{\circ}$ .<sup>70</sup>
332. 4,3-HO(MeO)C<sub>6</sub>H<sub>3</sub>COMe, m.  $209^{\circ}$  dec.<sup>70</sup>
333. "Vanillalacetone," m.  $95^{\circ}$ .<sup>406</sup>
334. Dodecylvanillyl ether, m.  $128^{\circ}$ .<sup>540</sup>
335. *p*-Nicotinoyloxyacetophenone, m.  $230^{\circ}$ .<sup>596</sup>
336. *p*-Isonicotinoyloxyacetophenone, m.  $217-20^{\circ}$ .<sup>596</sup>
337. *p*-ClC<sub>6</sub>H<sub>4</sub>COMe, m.  $201^{\circ}$ ,<sup>351</sup>  $206^{\circ}$ .<sup>406</sup>
338. *p*-BrC<sub>6</sub>H<sub>4</sub>COMe, m.  $203^{\circ}$ .<sup>406</sup>
339. *m*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>COMe, m.  $228-30^{\circ}$ ,<sup>291</sup>  $237^{\circ}$ .<sup>406</sup>
340. *p*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>COMe, m.  $247^{\circ}$ ,<sup>406</sup>  $244^{\circ}$ ,<sup>351</sup>  $240^{\circ}$  dec.,<sup>349d</sup>  $223^{\circ}$  dec.<sup>331</sup>
341. 3,5-(O<sub>2</sub>N)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>COMe, m.  $258^{\circ}$ .<sup>351</sup>
342. *m*-H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>COMe, m.  $154^{\circ}$ ,<sup>291</sup>  $152-4^{\circ}$ .<sup>406</sup>
343. *p*-H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>COMe, m.  $193-5^{\circ}$ ,<sup>291</sup>  $190^{\circ}$ ,<sup>406</sup>  $185^{\circ}$ ,<sup>349d</sup>  $179^{\circ}$ .<sup>329</sup>
344. *p*-AcNHC<sub>6</sub>H<sub>4</sub>COMe, m.  $245^{\circ}$  dec.,<sup>291</sup>  $233^{\circ}$ ,<sup>406</sup>  $223.5^{\circ}$ .<sup>349d</sup>

345. *p*-EtCONHC<sub>6</sub>H<sub>4</sub>COMe, m. 236°. <sup>406</sup>
346. *p*-(*p*-H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>SO<sub>2</sub>NH)C<sub>6</sub>H<sub>4</sub>COMe, m. 240°. <sup>596</sup>
347. *p*-(*p*-AcNHC<sub>6</sub>H<sub>4</sub>SO<sub>2</sub>NH)C<sub>6</sub>H<sub>4</sub>COMe, m. 223–5°. <sup>596</sup>
348. *p*-Nicotinoylaminoacetophenone, m. 247°. <sup>596</sup>
349. *p*-Isonicotinoylaminoacetophenone, m. 256°. <sup>596</sup>
350. *p*-MeC<sub>6</sub>H<sub>4</sub>COMe, m. 162°, <sup>406</sup> 159°. <sup>87</sup>
351. 3,4-Me(PhCH<sub>2</sub>O)C<sub>6</sub>H<sub>3</sub>COMe, m. 174°. <sup>435</sup>
352. 2,5,4-Me(Me<sub>2</sub>CH)(HO)C<sub>6</sub>H<sub>2</sub>COMe, m. 213°, <sup>291</sup> 210°. <sup>608</sup>
353. 2,5,4-Me(Me<sub>2</sub>CH)(MeO)C<sub>6</sub>H<sub>2</sub>COMe, m. 168°. <sup>608</sup>
354. PhCH<sub>2</sub>COMe, m. 155°, <sup>406</sup> 120–30°. <sup>235</sup>
355. *p*-MeOC<sub>6</sub>H<sub>4</sub>CHOHCOMe, m. 199°. <sup>617</sup>
356. 2,4-HO(MeO)C<sub>6</sub>H<sub>3</sub>CH<sub>2</sub>CH<sub>2</sub>COMe, m. 135–7°. <sup>521</sup>
357. 4,2-HO(MeO)C<sub>6</sub>H<sub>3</sub>CH<sub>2</sub>CH<sub>2</sub>COMe, m. 116–18°. <sup>521</sup>
358. PhCH:CHCOMe, m. 147°, <sup>351</sup> 140°; <sup>329</sup> anti form m. 147°;  
syn form m. 131.5°. <sup>406</sup>
359. *o*-HOC<sub>6</sub>H<sub>4</sub>CH:CHCOMe, m. 186°, <sup>329</sup> 195°. <sup>131d</sup>
360. *p*-AcOC<sub>6</sub>H<sub>4</sub>CH:CHCOMe, m. 158°. <sup>131d</sup>
361. MeOC<sub>6</sub>H<sub>4</sub>CH:CHCOMe, m. 173°. <sup>329</sup>
362. *m*-MeOC<sub>6</sub>H<sub>4</sub>CH:CHCOMe, m. 170°. <sup>131d</sup>
363. *p*-MeOC<sub>6</sub>H<sub>4</sub>CH:CHCOMe, m. 196–8°, <sup>406</sup> 192°, <sup>131d</sup> 187°  
dec. <sup>70</sup>
364. *p*-AllylOC<sub>6</sub>H<sub>4</sub>CH:CHCOMe, m. 145°. <sup>131d</sup>
365. *p*-PhOC<sub>6</sub>H<sub>4</sub>CH:CHCOMe, m. 166°. <sup>131d</sup>
366. 2,3-(MeO)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>CH:CHCOMe, m. 188°. <sup>351</sup>
367. 2,4-(MeO)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>CH:CHCOMe, m. 168–70°. <sup>521</sup>
368. 2,4-HO(MeO)C<sub>6</sub>H<sub>3</sub>CH:CHCOMe, m. 120–2°. <sup>521</sup>
369. 4,2-HO(MeO)C<sub>6</sub>H<sub>3</sub>CH:CHCOMe, m. 166.5°. <sup>521</sup>
370. *o*-ClC<sub>6</sub>H<sub>4</sub>CH:CHCOMe, m. 185°, <sup>351</sup> 166°. <sup>70</sup>
371. *p*-ClC<sub>6</sub>H<sub>4</sub>CH:CHCOMe, m. 191°, <sup>131d</sup> 188°. <sup>70</sup>
372. 2,4-Cl<sub>2</sub>C<sub>6</sub>H<sub>3</sub>CH:CHCOMe, m. 196°. <sup>70</sup>
373. 3,4-Cl<sub>2</sub>C<sub>6</sub>H<sub>3</sub>CH:CHCOMe, m. 188°. <sup>70</sup>
374. *p*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CH:CHCOMe, m. 250°, <sup>351</sup> 274° dec., <sup>181d</sup>  
240°. <sup>349b</sup>
375. *p*-H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CH:CHCOMe, m. 198° dec., <sup>131d</sup> 191° dec. <sup>349b</sup>
376. *p*-AcNHC<sub>6</sub>H<sub>4</sub>CH:CHCOMe, m. 223° dec. <sup>349b</sup>
377. *p*-Me<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CH:CHCOMe, m. 208°. <sup>131d</sup>
378. *p*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CH:CBrCOMe, m. 191° dec. <sup>349b</sup>
379. *p*-PhC<sub>6</sub>H<sub>4</sub>COMe, m. 264°, <sup>351</sup> 232°. <sup>406</sup>
380. *p*-(*p*-MeOC<sub>6</sub>H<sub>4</sub>)C<sub>6</sub>H<sub>4</sub>COMe, m. 280° dec. <sup>102</sup>
381. *p*-(*p*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>)C<sub>6</sub>H<sub>4</sub>COMe, m. 265°. <sup>351</sup>



382.  $\alpha$ -C<sub>10</sub>H<sub>7</sub>COMe, m. 183°,<sup>406</sup> 175°.<sup>87</sup>  
383.  $\beta$ -HOC<sub>10</sub>H<sub>6</sub>COMe, m. 105°.<sup>406</sup>  
384. 6,2-MeSC<sub>10</sub>H<sub>6</sub>COMe, m. 201°.<sup>538</sup>  
385. Propiophenone, m. 114°,<sup>87, 291</sup> 115–17°;<sup>235</sup> antiform, m. 107–10°; synform, m. 93.5°.<sup>406</sup>  
386. *p*-HOC<sub>6</sub>H<sub>4</sub>COEt, m. 147°,<sup>406</sup> 86°,<sup>291</sup> 200° dec.<sup>70</sup>  
387. *p*-MeOC<sub>6</sub>H<sub>4</sub>COEt, m. 105°,<sup>406</sup> 145°,<sup>331</sup> 136°.<sup>291</sup>  
388. *p*-EtOC<sub>6</sub>H<sub>4</sub>COEt, m. 127°.<sup>291</sup>  
389. *p*-Me<sub>2</sub>CHCH<sub>2</sub>CH<sub>2</sub>OC<sub>6</sub>H<sub>4</sub>COEt, m. 205° dec., 35°.<sup>70</sup>  
390. *p*-PhCH<sub>2</sub>OC<sub>6</sub>H<sub>4</sub>COEt, m. 156°,<sup>291</sup> 146°,<sup>435</sup> 200–2° dec.<sup>70</sup>  
391. *p*-MeOC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>OC<sub>6</sub>H<sub>4</sub>COEt, m. 209° dec.<sup>70</sup>  
392. *p*-(*p*-MeC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>O)C<sub>6</sub>H<sub>4</sub>COEt, m. 109°,<sup>435</sup> 74°.<sup>70</sup>  
393. 3,4-(MeO)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>COEt, m. 160°.<sup>70</sup>  
394. *p*-ClC<sub>6</sub>H<sub>4</sub>COEt, m. 181°,<sup>406</sup> 176°.<sup>351</sup>  
395. *p*-H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>COEt, m. 90–2°.<sup>406</sup>  
396. 2,4-Me(HO)C<sub>6</sub>H<sub>3</sub>COEt, m. 113–15°.<sup>406</sup>  
397. 2,4-Me(MeO)C<sub>6</sub>H<sub>3</sub>COEt, m. 147–9°.<sup>406</sup>  
398. 3,4-Me(MeO)C<sub>6</sub>H<sub>3</sub>COEt, m. 178–81°.<sup>406</sup>  
399. 6,2-MeSC<sub>10</sub>H<sub>6</sub>COEt, m. 180°.<sup>538</sup>  
400. Butyrophenone, m. 92°,<sup>87</sup> 70–2°.<sup>406</sup>  
401. *i*-Butyrophenone, m. 160–5°.<sup>87</sup>  
402. Valerophenone, m. 80–2°.<sup>406</sup>  
403. HOCH<sub>2</sub>COPh, m. 171°.<sup>235</sup>  
404. HOCHMeCOPh, m. 143°.<sup>617</sup>  
405. HOCHMeCOC<sub>6</sub>H<sub>4</sub>Me-*p*, m. 166°.<sup>617</sup>  
406. EOCCOC<sub>6</sub>H<sub>4</sub>OMe-*p*, m. 146°.<sup>253b</sup>  
407. *p*-Acetamidobenzoyl formic acid, m. 190°.<sup>481</sup>  
408. *p*-Acetamidobenzoyl pyruvic acid, m. 178–80°.<sup>481</sup>  
409. *p*-AcNHC<sub>6</sub>H<sub>4</sub>COCH<sub>2</sub>COCOOEt, m. 125°.<sup>481</sup>  
410. *p*-Nitrobenzoyl acetic acid, Et ester, m. 168°.<sup>481</sup>  
411. *p*-Aminobenzoyl acetic acid, Et ester, m. 145°.<sup>481</sup>  
412. HOOCCH<sub>2</sub>CH<sub>2</sub>COPh, m. 171°.<sup>351</sup>  
413. *p*-Hydroxybenzoyl propionic acid, m. 192°.<sup>481</sup>  
414. AcOCH<sub>2</sub>COC<sub>6</sub>H<sub>4</sub>NO<sub>2</sub>-*p*, m. 212°.<sup>351</sup>  
415. H<sub>2</sub>NCH<sub>2</sub>COC<sub>6</sub>H<sub>4</sub>NO<sub>2</sub>-*p*, HCl, m. 222°.<sup>351</sup>  
416. AcNHCH<sub>2</sub>COC<sub>6</sub>H<sub>4</sub>NO<sub>2</sub>-*p*, m. 240°.<sup>351</sup>  
417. Cl<sub>2</sub>CHCONHCH<sub>2</sub>COC<sub>6</sub>H<sub>4</sub>NO<sub>2</sub>-*p*, m. 196°.<sup>351</sup>  
418. Me<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>COC<sub>6</sub>H<sub>4</sub>NO<sub>2</sub>-*p*, HCl, m. 153°.<sup>351</sup>  
419. *p*-BrC<sub>6</sub>H<sub>4</sub>COCH<sub>2</sub>SC<sub>6</sub>H<sub>4</sub>NO<sub>2</sub>-*p*, m. 173°.<sup>473</sup>  
420. *p*-BrC<sub>6</sub>H<sub>4</sub>COCH<sub>2</sub>SO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>NO<sub>2</sub>-*p*, m. 222°.<sup>473</sup>

421.  $p\text{-ClC}_6\text{H}_4\text{COCH}_2\text{SO}_2\text{C}_6\text{H}_4\text{NO}_2\text{-}p$ , m.  $202^\circ$ .<sup>473</sup>  
422.  $p\text{-MeC}_6\text{H}_4\text{COCH}_2\text{SO}_2\text{C}_6\text{H}_4\text{NO}_2\text{-}p$ , m.  $197^\circ$ .<sup>473</sup>  
423. Benzophenone, m.  $171^\circ$ .<sup>331</sup>  
424.  $\text{PhCH}_2\text{COPh}$ , m.  $121^\circ$ .<sup>406</sup>  
425.  $\text{PhCH(OH)COPh}$ , m.  $139^\circ$ ,<sup>406</sup>  $176\text{--}8^\circ$ .<sup>235</sup>  
426. 4-Fluoro-2-methyl- $\alpha$ -phenylacetophenone, m.  $138^\circ$ .<sup>539</sup>  
427.  $\text{PhCH:CHCOPh}$ , m.  $143^\circ$ .<sup>131g</sup>  
428.  $\text{PhCH:CHCOCH}_2\text{Ph}$ , m.  $135^\circ$ .<sup>414a</sup>  
429.  $p\text{-HOC}_6\text{H}_4\text{CH:CHCOPh}$ , m.  $144^\circ$ .<sup>131g</sup>  
430.  $p\text{-AcOC}_6\text{H}_4\text{CH:CHCOPh}$ , m.  $186^\circ$ .<sup>131g</sup>  
431.  $o\text{-MeOC}_6\text{H}_4\text{CH:CHCOPh}$ , m.  $174^\circ$ .<sup>131g</sup>  
432.  $m\text{-MeOC}_6\text{H}_4\text{CH:CHCOPh}$ , m.  $146^\circ$ .<sup>131g</sup>  
433.  $p\text{-MeOC}_6\text{H}_4\text{CH:CHCOPh}$ , m.  $142^\circ$ ; recrystallized from  $\text{AcOH}$ , m.  $190^\circ$ .<sup>131g</sup>  
434.  $p\text{-EtOC}_6\text{H}_4\text{CH:CHCOPh}$ , m.  $157^\circ$ .<sup>131g</sup>  
435.  $p\text{-PrOC}_6\text{H}_4\text{CH:CHCOPh}$ , m.  $164^\circ$ .<sup>131g</sup>  
436.  $p\text{-BuOC}_6\text{H}_4\text{CH:CHCOPh}$ , m.  $150^\circ$ .<sup>131g</sup>  
437.  $p\text{-Allyl OC}_6\text{H}_4\text{CH:CHCOPh}$ , m.  $159^\circ$ .<sup>131g</sup>  
438.  $p\text{-PhOC}_6\text{H}_4\text{CH:CHCOPh}$ , m.  $171^\circ$ .<sup>131g</sup>  
439. 3,4-( $\text{MeO}$ ) $_2\text{C}_6\text{H}_3\text{CH:CHCOPh}$ , m.  $165^\circ$ .<sup>131g</sup>  
440.  $m\text{-HOOCCH}_2\text{OC}_6\text{H}_4\text{CH:CHCOPh}$ , m.  $181^\circ$ .<sup>131g</sup>  
441.  $p\text{-HOOCCH}_2\text{OC}_6\text{H}_4\text{CH:CHCOPh}$ , m.  $208^\circ$ .<sup>131g</sup>  
442.  $p\text{-O}_2\text{NC}_6\text{H}_4\text{CH:CHCOPh}$ , m.  $185^\circ$ .<sup>131g</sup>  
443.  $p\text{-AcNHC}_6\text{H}_4\text{CH:CHCOPh}$ , m.  $205^\circ$ .<sup>131g</sup>  
444. 3,5- $\text{Br}_2\text{C}_6\text{H}_3\text{CH:CHCOPh}$ , m.  $227^\circ$ .<sup>131g</sup>  
445.  $p\text{-EtSO}_2\text{C}_6\text{H}_4\text{CH:CHCOPh}$ , m.  $211^\circ$ .<sup>131g</sup>  
446.  $p\text{-HOCC}_6\text{H}_4\text{CH:CHCOPh}$ , m.  $253^\circ$ .<sup>131g</sup>  
447.  $p\text{-HOCCCH:CHC}_6\text{H}_4\text{CH:CHCOPh}$ , m.  $258^\circ$ .<sup>131g</sup>  
448.  $p\text{-MeOC}_6\text{H}_4\text{CH:CHCOC}_6\text{H}_4\text{OMe-}p$ , m.  $180^\circ$ .<sup>131g</sup>  
449.  $p\text{-MeOC}_6\text{H}_4\text{CH:CHCOC}_6\text{H}_4\text{Cl-}p$ , m.  $217^\circ$ .<sup>131g</sup>  
450.  $p\text{-HOCC}_6\text{H}_4\text{CH:CHCOC}_6\text{H}_4\text{Cl-}p$ , m.  $263^\circ$ .<sup>131g</sup>  
451.  $p\text{-MeOC}_6\text{H}_4\text{CH:CHCOC}_6\text{H}_4\text{Br-}p$ , m.  $223^\circ$ .<sup>131g</sup>  
452.  $p\text{-MeOC}_6\text{H}_4\text{CH:CHCOC}_6\text{H}_4\text{SMe-}p$ , m.  $155^\circ$ .<sup>131g</sup>  
453.  $\text{PhCH:CHCOC}_6\text{H}_4\text{COOH-}p$ , m.  $224^\circ$ .<sup>131g</sup>  
454.  $p\text{-MeOC}_6\text{H}_4\text{CH:CHCOC}_6\text{H}_4\text{COOH-}p$ , m.  $224^\circ$ .<sup>131g</sup>  
455.  $p\text{-MeOC}_6\text{H}_4\text{CH:CHCOC}_6\text{H}_4\text{Me-}p$ , m.  $199^\circ$ .<sup>131g</sup>  
456.  $p\text{-EtOC}_6\text{H}_4\text{CH:CHCOC}_6\text{H}_4\text{Me-}p$ , m.  $55^\circ$ .<sup>131g</sup>  
457.  $p\text{-MeOC}_6\text{H}_4\text{CH:CHCOC}_{10}\text{H}_7\text{-}\beta$ , m.  $117^\circ$ .<sup>131g</sup>  
458.  $p\text{-MeOC}_6\text{H}_4\text{CH:CHCO thienyl-}\alpha$ , m.  $173^\circ$ .<sup>131g</sup>  
459. 5-Cinnamoyl-8-hydroxyquinoline, m.  $234^\circ$ .<sup>131g</sup>

460. Benzoquinone guanyllhydrazone, m. 192–4° dec.<sup>374</sup>  
 461. Fluorenone, m. 237°.<sup>406</sup>  
 462. Xanthone, m. 182°.<sup>406</sup>  
 463. Acenaphthenone, m. 228°.<sup>177b</sup>

#### DIKETO COMPOUNDS

464. (MeCO)<sub>2</sub>, m. 136–41°.<sup>406</sup>  
 465. (PhCO)<sub>2</sub>, m. 96°; <sup>406</sup>  $\text{Ph}\overline{\text{C}}:\text{NN}:\text{C}(\text{SH})\text{N}:\text{C}-\text{Ph}$ , m. 220°.<sup>22</sup>  
 466. Anisil, mono, m. 228°.<sup>177b</sup>  
 467. Acenaphthenequinone, mono, m. 224°.<sup>177b</sup>  
 468. Naphthazarin, mono, 160° dec.<sup>177b</sup>  
 469. Vitamin K<sub>3</sub> (2-Me-1,4-naphthoquinone), m. 231°.<sup>406</sup>  
 470. Phthiacol (2-Me-3-hydroxy-1,4-naphthoquinone), m. 180°.<sup>406</sup>

#### HETEROCYCLIC ALDEHYDES AND KETONES

471. Furfural, m. 183°.<sup>406</sup> 152–4°.<sup>6</sup> 149°.<sup>87</sup> 148°.<sup>329</sup>  
 472. 5-Nitrofurfural, m. 237°.<sup>406</sup>  
 473. 5-Hydroxymethyl furfural, m. 190°.<sup>177a</sup>  
 474. 4-Acetyl-5-methyl furfural, m. 213–15°.<sup>408</sup>  
 475. 4-Carboxy-5-methyl furfural, m. 245–7° dec.<sup>403</sup>  
 476. 4-Carbethoxy-5-methyl furfural, m. 215–18°.<sup>403</sup>  
 477. Furyl aceolien, m. 158°.<sup>406</sup>  
 478. 2-Formyl coumarone, dec. 205°.<sup>255</sup>  
 479. 5-Nitro-2-formylcoumarone, m. 251°.<sup>255</sup>  
 480. 2-Thienylaldehyde, m. 186°.<sup>6</sup>  
 481. 5-Chloro-2-thienylaldehyde, m. 165°.<sup>78</sup> 158°.<sup>94</sup>  
 482. 5-Bromo-2-thienylaldehyde, m. 182–4°.<sup>78</sup> 175°.<sup>94</sup>  
 483. 5-Nitro-2-thienylaldehyde, m. 252–5° dec.<sup>78</sup> 245–50°.<sup>94</sup>  
 484. 5-Acetamido-2-thienylaldehyde, m. 231–3°.<sup>78</sup>  
 485. 5-Methylmercapto-2-thienylaldehyde, m. 145°.<sup>102</sup>  
 486. 3-Methyl-2-thienylaldehyde, m. 185–7°.<sup>78</sup>  
 487. 3-Methyl-5-nitro-2-thienylaldehyde, m. 212–15°.<sup>78</sup>  
 488. 5-Methyl-2-thienylaldehyde, m. 161°.<sup>78</sup> 142–4°.<sup>94</sup>  
 489. 5-Propyl-2-thienylaldehyde, m. 119°.<sup>541</sup>  
 490. 5-*t*-Butyl-2-thienylaldehyde, m. 183°.<sup>78</sup> 197°.<sup>615</sup>  
 491. 5-Dodecyl-2-thienylaldehyde, m. 95°.<sup>541</sup>  
 492. 5-Tetradecyl-2-thienylaldehyde, m. 97°.<sup>541</sup>  
 493. 5-Phenyl-2-thienylaldehyde, m. about 209°.<sup>553</sup>

494. 5-*m*-Chlorophenyl-2-thienylaldehyde, m. 186°. <sup>553</sup>  
495. 5-*m*-Tolyl-2-thienylaldehyde, m. 191°. <sup>553</sup>  
496.  $\beta$ -2-thienylacrolein, m. 102°. <sup>77a</sup>  
497. 5-Formyl-2-styrylthiophene, m. 187°. <sup>597</sup>  
498. 1-(5-Formyl-2-thienyl)-1-phenyl-2-(*p*-chlorophenyl) ethylene, m. 212°. <sup>597</sup>  
499. 3,5-Di-*p*-methoxyphenyl-2-selenophenealdehyde, m. 244°. <sup>553</sup>  
500. 3,5-Di-*p*-tolyl-2-selenophenealdehyde, m. 192°. <sup>553</sup>  
501. 3-Thienylaldehyde, m. 152°. <sup>78</sup>  
502. 2-Chloro-3-thienylaldehyde, m. 196–8° dec. <sup>78</sup>  
503. 2,5-Dichloro-3-thienylaldehyde, m. 233° dec. <sup>78</sup>  
504. 2-Bromo-3-thienylaldehyde, m. 192–4° dec. <sup>78</sup>  
505. 3-Thianaphthencarboxaldehyde, m. 218°. <sup>32d</sup>  
506. 3-Hydroxythianaphthene, m. 193°. <sup>32d</sup>  
507. 2-Pyrrylaldehyde, m. 204°, <sup>253a</sup> 195–7°. <sup>6</sup>  
508. 5-Methyl-4-carboxy-2-pyrrylaldehyde, dec. 235–45°. <sup>403</sup>  
509. 5-Methyl-4-acetyl-2-pyrrylaldehyde, dec. 240–50°. <sup>403</sup>  
510. 1-Methyl-2-pyrrylaldehyde, m. 163°. <sup>213a</sup>  
511. 2-Indolylaldehyde, m. 229°. <sup>118</sup>  
512. 1,3,3-Trimethylindolylaldehyde, m. 176°. <sup>32d</sup>  
513. 1,3,3-Trimethyl-5-methoxyindolylaldehyde, m. 178°. <sup>32d</sup>  
514. 3-Indolylaldehyde, m. 265°, <sup>291</sup> 232°. <sup>232</sup>  
515. 2-Oxo-3-indolylaldehyde, m. 268° dec. <sup>520</sup>  
516. 1-Methyl-3-indolylaldehyde, m. 177°. <sup>32d</sup>  
517. 2-Methyl-3-indolylaldehyde, m. 213°. <sup>32d</sup>  
518. 2-Methyl-2,3-dihydroindolyl-5-aldehyde, m. 176°. <sup>32d</sup>  
519. 2-Pyridylaldehyde, m. 209°, <sup>32c</sup> 207°. <sup>229</sup>  
520. 5-Chloro-2-pyridylaldehyde, m. 154°. <sup>213a</sup>  
521. 6-Methyl-2-pyridylaldehyde, m. 204°. <sup>213a</sup>  
522. 1-Methyl-1,2-dihydro-2-pyridylaldehyde, m. 198° dec. <sup>372</sup>  
523. 3-Pyridylaldehyde (nicotinealdehyde), m. 226°, <sup>117</sup> 227–30° dec., <sup>280</sup> 224° dec., <sup>445</sup> 223° dec., <sup>177a</sup> 216°, <sup>32c</sup> 213°; <sup>6</sup> HCl-SnCl<sub>2</sub>, m. 219°. <sup>177a</sup>  
524. 5-Bromo-3-pyridylaldehyde, m. 261–3°. <sup>213a</sup>  
525. 5-Amino-3-pyridylaldehyde, m. 225°. <sup>213a</sup>  
526. 5-Acetamido-3-pyridylaldehyde, m. 224–6°. <sup>213a</sup>  
527. 6-Amino-3-pyridylaldehyde, m. 219–21°. <sup>213a</sup>  
528. 6-Dimethylamino-3-pyridylaldehyde, m. 230–2°. <sup>253a</sup>  
529. 6-Diethylamino-3-pyridylaldehyde, m. 214–16°. <sup>253a</sup>

530. 2-Methyl-3-pyridylaldehyde, m.  $242^{\circ}$ .<sup>117</sup>  
531. 6-Methyl-3-pyridylaldehyde, m.  $234^{\circ}$ .<sup>213a</sup>  
532. 2-Hydroxy-4-ethoxymethyl-6-methyl-3-pyridylaldehyde,  
m.  $> 250^{\circ}$ .<sup>177a</sup>  
533. 4,6-Dimethyl-5-hydroxy-3-pyridylaldehyde, m.  $213^{\circ}$ .<sup>177a</sup>  
534. 4-Pyridylaldehyde (isonicotinaldehyde), m.  $226-8^{\circ}$  dec.,<sup>32c</sup>  
 $224-6^{\circ}$ ,<sup>411</sup>  $224-6^{\circ}$  dec.,<sup>1311</sup>  $221^{\circ}$  dec.,<sup>445</sup>  $219-20^{\circ}$  dec.,<sup>149a</sup>  
 $219^{\circ}$ ,<sup>149b</sup>  $215-17^{\circ}$ ,<sup>609</sup>  $208^{\circ}$  dec.; mono HCl salt, m.  $269^{\circ}$ ;  
EtSO<sub>3</sub>H·H<sub>2</sub>O salt, m.  $208^{\circ}$  dec.<sup>149a</sup>  
535. 2-Methyl-3-hydroxy-5-hydroxymethyl-isonicotinaldehyde,  
m.  $194^{\circ}$  dec.<sup>177b</sup>  
536. Picolinaldehyde, m.  $210^{\circ}$ ,<sup>445</sup>  $205^{\circ}$ ; mono HCl salt, m.  $230^{\circ}$   
dec.<sup>149a</sup>  
537. 2,3-Diformyl pyridine, dec. above  $350^{\circ}$ .<sup>445</sup>  
537.5. 2,4-Diformyl pyridine, m.  $240.5^{\circ}$ .<sup>445</sup>  
538. 2,5-Diformyl pyridine, m.  $239^{\circ}$ .<sup>445</sup>  
539. 2,6-Diformylpyridine, m.  $242^{\circ}$ .<sup>445</sup>  
540. 2,4,6-Triformylpyridine, m.  $166^{\circ}$ .<sup>445</sup>  
541. 5-Hydroxy-6-methyl-cinchomeronaldehyde, m.  $144-6^{\circ}$   
dec.<sup>177a</sup>  
542. Nicotinic acid, m.  $184^{\circ}$ .<sup>149a</sup>  
543. Isonicotinic acid, m.  $280^{\circ}$  dec.<sup>149a</sup>  
544. 2-Quinolinecarboxaldehyde, m.  $138^{\circ}$ .<sup>250</sup>  
545. 6-Quinolinecarboxaldehyde, m.  $219^{\circ}$ .<sup>32d</sup>  
546. 1,2,3,4-Tetrahydroquinoline-6-carboxaldehyde, m.  $219^{\circ}$ .<sup>32d</sup>  
547. 1-Methyl-1,2,3,4-tetrahydroquinoline-6-carboxaldehyde, m.  
 $219^{\circ}$ .<sup>32d</sup>  
548. 5-Formyl-8-quinolinol, hemihydrate, m.  $265^{\circ}$ .<sup>586</sup>  
549. 9-Acridinecarboxaldehyde, m.  $235^{\circ}$ .<sup>32d</sup>  
550. 4-Imidazolylaldehyde, m.  $208^{\circ}$ .<sup>213a</sup>  
551. Antipyralsaldehyde, m.  $227^{\circ}$ ,<sup>177a</sup>  $226^{\circ}$ .<sup>32d</sup>  
552. 1,5-Dimethyl-2-ethoxy-3-pyrazolone-4-aldehyde, m.  $233-5^{\circ}$ .<sup>287</sup>  
553. 1,5-Dimethyl-2-phenyl-3-pyrazolone-4-aldehyde, m.  $247^{\circ}$ .<sup>287</sup>  
554. 1-Methyl-2,5-diphenyl-3-pyrazolone-4-aldehyde, m.  $233-5^{\circ}$ .<sup>287</sup>  
555. Pyrazinaldehyde, m.  $237-9^{\circ}$  dec.<sup>279</sup>  
556. 2-Mercapto-6-hydroxy-4-pyrimidylaldehyde, m.  $> 380^{\circ}$ .<sup>213a</sup>  
557. 2-Ethylmercapto-6-hydroxy-4-pyrimidylaldehyde, m.  $260-2^{\circ}$ .<sup>213a</sup>

558. 1-Methyl-6-aminopyrimidylaldehyde, m.  $260^{\circ}$  dec.<sup>544</sup>  
559. 1,4,6-Trimethyl-2-pyrimidylaldehyde, m.  $202^{\circ}$  dec.<sup>372</sup>  
560. 4,7-Phenanthroline-1-carboxaldehyde, m.  $230^{\circ}$  dec.<sup>127</sup>  
561. 1,7-Phenanthroline-4-carboxaldehyde, m.  $252^{\circ}$  dec.<sup>127</sup>  
562. 1,7-Phenanthroline-8-carboxaldehyde, m.  $240^{\circ}$  dec.<sup>127</sup>  
563. 6,7-Dimethyl-9-formylmethylisoalloxazinehydrate, m.  $237^{\circ}$ .<sup>130</sup>  
564. 5-Phenyl-4-thiazolyl aldehyde, m.  $214-16^{\circ}$ .<sup>213a</sup>  
565. 1,5-Diphenyl-4-thiazolyl aldehyde, m.  $226^{\circ}$ .<sup>213a</sup>  
566. 3,5-Diphenyl-4-thiazolyl aldehyde, m.  $239-41^{\circ}$ .<sup>213a</sup>  
567. 4-Thiazolyl aldehyde, m.  $190^{\circ}$ ,<sup>213a</sup>  $182^{\circ}$ .<sup>291</sup>  
568. 2-Acetamido-4-thiazolyl aldehyde, m.  $260^{\circ}$ .<sup>460</sup>  
569. 2-Benzamido-4-thiazolyl aldehyde, m.  $> 360^{\circ}$ .<sup>460</sup>  
570. 2-Phenylacetamido-4-thiazolyl aldehyde, m.  $280^{\circ}$ .<sup>460</sup>  
571. *p*-Aminophenylsulfonamido-4-thiazolyl aldehyde, m.  $218-20^{\circ}$ .<sup>460</sup>  
572. *p*-Acetamidophenylsulfonamido-4-thiazolyl aldehyde, m.  $245^{\circ}$  dec.<sup>460</sup>  
573. 2-Anilino-4-thiazolyl aldehyde, m.  $245^{\circ}$ .<sup>460</sup>  
574. 2-Methyl-4-thiazolyl aldehyde, m.  $240^{\circ}$  dec.<sup>460</sup>  
575. 3-Phenyl-2-phenylimino-4-thiazoline-5-carboxaldehyde, m.  $207^{\circ}$  dec.<sup>460</sup>  
576. 2,3-Dihydro-3-methyl-2-thiazolyl aldehyde, m.  $214^{\circ}$  dec.<sup>372</sup>  
577. 5-Thiazolyl aldehyde, m.  $224^{\circ}$ ,<sup>213a</sup>  $229^{\circ}$ .<sup>117</sup>  
578. 4-Methyl-5-thiazolyl aldehyde, m.  $233^{\circ}$ ,<sup>117</sup>  $228-30^{\circ}$ .<sup>213a</sup>  
579. 2-Benzothiazolyl aldehyde, dec.  $264^{\circ}$ .<sup>634</sup>  
580. 6-Methoxy-2-benzothiazolyl aldehyde, m.  $253^{\circ}$ .<sup>634</sup>  
581. 6-Chloro-2-benzothiazolyl aldehyde, dec.  $263^{\circ}$ .<sup>634</sup>  
582. 5-Nitro-2-benzothiazolyl aldehyde, dec.  $276^{\circ}$ .<sup>634</sup>  
583. 6-Nitro-2-benzothiazolyl aldehyde, m.  $287-9^{\circ}$ .<sup>634</sup>  
584. *p*-HOCC<sub>6</sub>H<sub>4</sub> ether of 2-benzothiazolyl carbinol, m.  $212^{\circ}$  dec.<sup>634</sup>  
585. AcC<sub>6</sub>H<sub>4</sub> ether of 2-benzothiazolyl carbinol, m.  $216-18^{\circ}$ .<sup>634</sup>  
586. 6-Methyl-2-benzothiazolyl aldehyde, m.  $265^{\circ}$  dec.<sup>634</sup>  
587. 2-Acetylfuron, m.  $142-4^{\circ}$ .<sup>6, 424</sup>  
588. 2-Butyrylfuron, m.  $127-9^{\circ}$ .<sup>6, 424</sup>  
589. 2,5-Dimethyl-3-acetylfuron, m.  $163^{\circ}$ .<sup>6, 424</sup>  
590. 5-Nitro-2-furyl-2-(1-dimethylamino)ethylketone, m.  $194^{\circ}$ .<sup>570</sup>  
591. Furoin, m.  $145^{\circ}$ .<sup>6</sup>

592. 2-Acetylthiophene, m.  $149^{\circ}$ ,<sup>6</sup>  $148^{\circ}$ ,<sup>78</sup>  $136^{\circ}$ ,<sup>351</sup>  $135^{\circ}$ .<sup>87</sup>  
 593. 5-Bromo-2-acetylthiophene, m.  $201^{\circ}$ .<sup>78</sup>  
 594. 5-Nitro-2-acetylthiophene, m.  $152^{\circ}$ .<sup>87</sup>  
 595. 5-Methyl-2-acetylthiophene, m.  $161-3^{\circ}$ .<sup>78</sup>  
 596. 5-Methyl-4-nitro-2-acetylthiophene, m.  $232-5^{\circ}$  dec.<sup>78</sup>  
 597. 5-(*t*-Butyl)-2-acetylthiophene, m.  $194^{\circ}$ .<sup>615</sup>  
 598. 5-(1,2-Diphenylethylene)-2-acetylthiophene, m.  $196^{\circ}$ .<sup>597</sup>  
 599. 5-(1-*p*-Tolyl-2-phenylethylene)-2-acetylthiophene, m.  $226^{\circ}$ .<sup>597</sup>  
 600. 5-(1-Phenyl-2-*p*-chlorophenylethylene)-2-acetylthiophene, m.  $196^{\circ}$  dec.<sup>597</sup>  
 601. 2-Propionylthiophene, m.  $128^{\circ}$ ,<sup>6</sup>  $117^{\circ}$ .<sup>291</sup>  
 602. 5-(*t*-Butyl)-2-propionylthiophene, m.  $187^{\circ}$ .<sup>615</sup>  
 603. 2-Butyrylthiophene, m.  $171^{\circ}$ .<sup>6</sup>  
 604. 2,5-Dimethyl-3-acetylthiophene, m.  $157^{\circ}$ .<sup>6</sup>  
 605. 3-(2-Thenoyl)propionic acid, m.  $198^{\circ}$ .<sup>473</sup>  
 606. 2,3-Thionaphthenequinone, m.  $203^{\circ}$ .<sup>32d</sup>  
 607. 2-Acetylpyrrole, m.  $185^{\circ}$ .<sup>177b</sup>  
 608. 2-Butyrylpyrrole, m.  $172.5^{\circ}$ .<sup>6</sup>  
 609. 2,5-Dimethyl-3-acetylpyrrole, m.  $200^{\circ}$ .<sup>6</sup>  
 610. Isatin, m.  $283^{\circ}$ ,<sup>291</sup>  $205^{\circ}$ ,<sup>329</sup>  $200^{\circ}$ .<sup>32d</sup>  
 611. 3-Indolylglyoxylic acid, m.  $204^{\circ}$ .<sup>118</sup>  
 612. 2-Acetylpyridine, m.  $158-60^{\circ}$ .<sup>6</sup>  
 613. 2-Pyridyl  $\text{COCH}\cdot\text{CO}\cdot\text{O}\cdot\text{CH}(\text{CH}_2\text{OEt})\cdot\text{CH}_2$ , m.  $125^{\circ}$ .<sup>630</sup>  
 614. 3-Acetylpyridine, m.  $225^{\circ}$ ,<sup>117</sup>  $217^{\circ}$  dec.;<sup>6</sup> HCl salt, m.  $161^{\circ}$ .<sup>149a</sup>  
 615. 2-Methyl-3-acetylpyridine, m.  $203^{\circ}$ .<sup>117</sup>  
 616. 6-Methyl-3-acetylpyridine, m.  $239^{\circ}$ .<sup>117</sup>  
 617. 3-Butyrylpyridine, m.  $184^{\circ}$ .<sup>6</sup>  
 618. 3-Benzoylpyridine, m.  $176^{\circ}$ .<sup>6</sup>  
 619. 4-Acetylpyridine, m.  $243^{\circ}$ ,<sup>291</sup>  $229-31^{\circ}$ ,<sup>411</sup>  $219-22^{\circ}$ ,<sup>6</sup> dec.  $230^{\circ}$ ; HCl salt, m.  $248^{\circ}$  dec.<sup>149a</sup>  
 620. 4-Propionylpyridine, m.  $190^{\circ}$ ,<sup>149a</sup>  $197^{\circ}$ .<sup>411</sup>  
 621. 4-Butyrylpyridine, m.  $176.5^{\circ}$ .<sup>149a</sup>  
 622. 2,2,6,6-Tetramethyl-4-piperidone, m.  $190-3^{\circ}$ .<sup>6</sup>  
 623. Nicotinoyl acetic acid, Et ester, m.  $217^{\circ}$ .<sup>481</sup>  
 624. Isonicotinoyl acetic acid, Et ester, m.  $162^{\circ}$ .<sup>481</sup>  
 625. Acetylpyrazine, m.  $227^{\circ}$  dec.<sup>279</sup>  
 626. Chloracetylpyrazine, m.  $222-4^{\circ}$ .<sup>279</sup>

627. 1,7-Phenanthrolene-5,6-dione, mono, m.  $> 300^{\circ}$ .<sup>548</sup>  
 628. 5-(C<sub>7</sub>H<sub>15</sub>COCH<sub>2</sub>)-2-thiohydantoin, m.  $177^{\circ}$ .<sup>517</sup>

Of H<sub>2</sub>NNHCSNHPh

629. Crotonaldehyde, m.  $141^{\circ}$ .<sup>478</sup>  
 630. Tetrolaldehyde, m.  $114-16^{\circ}$  dec.<sup>623</sup>  
 631. Benzaldehyde, m.  $191^{\circ}$ ,<sup>385</sup>  $189^{\circ}$ .<sup>467d, 548</sup>  
 632. *m*-HOC<sub>6</sub>H<sub>4</sub>CHO, m.  $194^{\circ}$ .<sup>564</sup>  
 633. *p*-HOC<sub>6</sub>H<sub>4</sub>CHO, m.  $225^{\circ}$ .<sup>564</sup>  
 634. *o*-MeOC<sub>6</sub>H<sub>4</sub>CHO, m.  $159^{\circ}$ .<sup>478</sup>  
 635. *m*-MeOC<sub>6</sub>H<sub>4</sub>CHO, m.  $154^{\circ}$ ,<sup>478</sup>  $153^{\circ}$ .<sup>564</sup>  
 636. *p*-MeOC<sub>6</sub>H<sub>4</sub>CHO, m.  $181^{\circ}$ ,<sup>478</sup>  $179^{\circ}$ .<sup>467d</sup> (anisaldehyde)  
 637. Piperonal, m.  $178^{\circ}$ .<sup>467d</sup>  
 638. Vanillin, m.  $180^{\circ}$ .<sup>467d</sup>  
 639. *o*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO, m.  $200^{\circ}$ .<sup>478</sup>  
 640. *m*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO, m.  $194^{\circ}$ .<sup>385</sup>  
 641. *p*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO, m.  $231^{\circ}$ .<sup>478</sup>  
 642. *p*-H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO, m.  $189^{\circ}$ .<sup>467d</sup>  
 643. *p*-AcNHC<sub>6</sub>H<sub>4</sub>CHO, m.  $201^{\circ}$ .<sup>177b</sup>  
 644. PhCH:CHCHO, m.  $176^{\circ}$ .<sup>385</sup>  
 645. Furfural, m.  $188^{\circ}$ ,<sup>478</sup>  $187^{\circ}$ .<sup>467d</sup>  
 646. 3-Pyridincarboxaldehyde, m.  $220^{\circ}$ .<sup>213a</sup>  
 647. CO·NPh·NMeCMe:(C(H)O), m.  $215-18^{\circ}$ .<sup>287</sup>  
 648. Acetone, m.  $132^{\circ}$ ,<sup>478</sup>  $130^{\circ}$ .<sup>438a</sup>  
 649. *c*-Pentanone, m.  $152^{\circ}$ .<sup>467d</sup>  
 650. *c*-Hexanone, m.  $114^{\circ}$ ,<sup>478</sup>  $111^{\circ}$ .<sup>467d</sup>  
 651. *c*-Heptanone, m.  $96^{\circ}$ .<sup>467d</sup>  
 652. *p*-ClC<sub>6</sub>H<sub>4</sub>COMe, m.  $180^{\circ}$ .<sup>467d</sup>  
 653. *D*-Carone, m.  $101^{\circ}$ .<sup>579</sup>  
 654. HON:CMe·COMe, m.  $201^{\circ}$ .<sup>552</sup>  
 655. (*D*-Camphor)quinone, m.  $184^{\circ}$ .<sup>148</sup>

Of H<sub>2</sub>NNHCSNHC<sub>6</sub>H<sub>4</sub>Me-*p*

656. Benzaldehyde, m.  $171^{\circ}$ ,<sup>164</sup>  $166^{\circ}$ .<sup>160c</sup>  
 657. Anisaldehyde, m.  $190^{\circ}$ .<sup>467d</sup>  
 658. *p*-H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO, m.  $173^{\circ}$ .<sup>467d</sup>  
 659. *D*-Mannose, m.  $187^{\circ}$ .<sup>347</sup>  
 660. *D*-Galactose, m.  $148^{\circ}$ .<sup>347</sup>  
 661. Furfural, m.  $162^{\circ}$ .<sup>467d</sup>



662. Acetone, m.  $142^{\circ}$ .<sup>52, 164</sup>  
 663. Butanone, m.  $75^{\circ}$ .<sup>52</sup>  
 664. *c*-Hexanone, m.  $125^{\circ}$ .<sup>52</sup>  
 665. Acetylacetone, m.  $100^{\circ}$ .<sup>52</sup>  
 666. Acetoacetic ester, m.  $107^{\circ}$ ,<sup>52</sup>  $87^{\circ}$ .<sup>160c</sup>  
 667. Benzoin, m.  $161^{\circ}$ .<sup>52</sup>  
 668. Carvone, m.  $147^{\circ}$ .<sup>52</sup>  
 669. Acetophenone, m.  $165^{\circ}$ ,<sup>52</sup>  $108^{\circ}$ .<sup>347</sup>  
 670. *p*-ClC<sub>6</sub>H<sub>4</sub>COMe, m.  $165^{\circ}$ .<sup>467d</sup>  
 671. Benzophenone, m.  $158^{\circ}$ .<sup>52</sup>

Of other H<sub>2</sub>NNHCSNHR

672. Methyl, PhCHO, m.  $170^{\circ}$ ,<sup>49</sup>  $160^{\circ}$ .<sup>385</sup>  
 673. 3-PyridylCHO, m.  $222^{\circ}$ .<sup>213a</sup>  
 674. Ethyl, 3-PyridylCHO, m.  $226^{\circ}$ .<sup>213a</sup>  
 675.  $\overline{\text{CO}\cdot\text{NPh}\cdot\text{NMe}\cdot\text{CMe}}\cdot\text{CCHO}$ , m.  $247^{\circ}$ .<sup>287</sup>  
 676. Allyl, PhCHO, m.  $125^{\circ}$ .<sup>386</sup>  
 677. *m*-HOC<sub>6</sub>H<sub>4</sub>CHO, m.  $144^{\circ}$ .<sup>422</sup>  
 678. *p*-HOC<sub>6</sub>H<sub>4</sub>CHO, m.  $182^{\circ}$ .<sup>422</sup>  
 679. 2,4-(HO)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>CHO, m.  $202^{\circ}$ .<sup>422</sup>  
 680. 3,4-(HO)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>CHO, m.  $200^{\circ}$ .<sup>422</sup>  
 681. Vanillin, m.  $134^{\circ}$ .<sup>422</sup>  
 682. PhCH:CHCHO, m.  $166^{\circ}$ .<sup>386</sup>  
 683. Isatin, m.  $211^{\circ}$ .<sup>422</sup>  
 684. 3-PyridylCHO, m.  $182^{\circ}$ .<sup>213a</sup>  
 685. 4-ThiazolylCHO, m.  $142^{\circ}$ .<sup>213a</sup>  
 686. Ph<sub>2</sub>CH, PhCHO, m.  $184^{\circ}$ .<sup>527</sup>

Of H<sub>2</sub>NNRCSNH<sub>2</sub>

687. Methyl, PhCH<sub>2</sub>COCOOH, m.  $250^{\circ}$ .<sup>83c</sup>  
 688. Phenyl, Me<sub>2</sub>CO, m.  $135^{\circ}$ .<sup>607</sup>  
 689. PhCHO, m.  $163^{\circ}$ .<sup>601</sup>  
 690. *m*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO, m.  $211^{\circ}$ .<sup>607</sup>  
 691. *p*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO, m.  $224^{\circ}$ .<sup>587</sup>  
 692. *p*-ClC<sub>6</sub>H<sub>4</sub>CHO, m.  $200.5^{\circ}$ .<sup>587</sup>  
 693. *p*-BrC<sub>6</sub>H<sub>4</sub>CHO, m.  $197.6^{\circ}$ .<sup>587</sup>  
 694. PhCO·COPh, m.  $185^{\circ}$ .<sup>607</sup>  
 695. Acetoacetic ester, m.  $142^{\circ}$ .<sup>607</sup>

Of  $\text{H}_2\text{NNR}_1\text{CSNHR}_2$ 

|      | $\text{R}_1$                          | $\text{R}_2$                 | Carbonyl Compound  |
|------|---------------------------------------|------------------------------|--|
| 696. | Methyl,                               | Phenyl,                      | $\text{PhCHO}$ , m. $132^\circ$ . <sup>62</sup> $131.5^\circ$ . <sup>320</sup> |
| 697. |                                       | Phenyl,                      | $o\text{-HOC}_6\text{H}_4\text{CHO}$ , m. $183^\circ$ . <sup>385</sup>         |
| 698. | Phenyl,                               | Methyl,                      | $\text{PhCHO}$ , m. $152^\circ$ . <sup>62</sup>                                |
| 699. |                                       | Phenyl,                      | $\text{Me}_2\text{CO}$ , m. $160^\circ$ . <sup>622</sup>                       |
| 700. |                                       | Phenyl,                      | $\text{MeCOEt}$ , m. $174^\circ$ . <sup>518</sup>                              |
| 701. |                                       | Phenyl,                      | $\text{PhCHO}$ , m. $171^\circ$ . <sup>57c</sup>                               |
| 702. |                                       | Benzyl,                      | $\text{Me}_2\text{CO}$ , m. $136^\circ$ . <sup>253</sup>                       |
| 703. | $m\text{-ClC}_6\text{H}_4$ ,          | Methyl,                      | $m\text{-O}_2\text{NC}_6\text{H}_4\text{CHO}$ , m. $224^\circ$ . <sup>63</sup> |
| 704. |                                       | Phenyl,                      | $\text{PhCHO}$ , m. $146^\circ$ . <sup>63</sup>                                |
| 705. | $m\text{-BrC}_6\text{H}_4$ ,          | Phenyl,                      | $\text{PhCHO}$ , m. $164^\circ$ . <sup>63</sup>                                |
| 706. | $m\text{-O}_2\text{NC}_6\text{H}_4$ , | Phenyl,                      | $\text{PhCHO}$ , m. $166^\circ$ . <sup>63</sup>                                |
| 707. | $m\text{-MeC}_6\text{H}_4$ ,          | Phenyl,                      | $\text{PhCHO}$ , m. $104^\circ$ . <sup>63</sup>                                |
| 708. |                                       | $p\text{-MeC}_6\text{H}_4$ , | $m\text{-O}_2\text{NC}_6\text{H}_4\text{CHO}$ , m. $198^\circ$ . <sup>63</sup> |
| 709. | $p\text{-MeC}_6\text{H}_4$ ,          | Phenyl,                      | $\text{Me}_2\text{CO}$ , m. $164^\circ$ . <sup>518</sup>                       |
| 710. | $\text{PhCH}_2$ ,                     | Methyl,                      | $\text{PhCHO}$ , m. $147^\circ$ . <sup>62</sup>                                |
| 711. |                                       | Allyl,                       | $\text{PhCHO}$ , m. $107^\circ$ . <sup>62</sup>                                |
| 712. |                                       | Phenyl,                      | $\text{PhCHO}$ , m. $132^\circ$ . <sup>62</sup>                                |
| 713. | $o\text{-MeOC}_6\text{H}_4$ ,         | Phenyl,                      | $m\text{-O}_2\text{NC}_6\text{H}_4\text{CHO}$ , m. $200^\circ$ . <sup>63</sup> |
| 714. | $\beta\text{-C}_{10}\text{H}_7$ ,     | Phenyl,                      | $\text{PhCHO}$ , m. $207^\circ$ . <sup>63</sup>                                |

Of  $\text{H}_2\text{NNR}_1\text{C(SMe):NR}_2$ 

|      |         |         |   |
|------|---------|---------|---|
| 715. | Phenyl, | Methyl, | $\text{PhCHO}$ , m. $137^\circ$ . <sup>62</sup> |
| 716. |         | Phenyl, | $\text{PhCHO}$ , m. $128^\circ$ . <sup>59</sup> |
| 717. | Benzyl, | Phenyl, | $\text{PhCHO}$ , m. $104^\circ$ . <sup>62</sup> |

Of  $\text{H}_2\text{NN:C(SR)NH}_2$ 

|      |         |  |
|------|---------|--|
| 718. | Methyl, | $\text{PhCHO}$ , m. $196^\circ$ . <sup>49</sup>                                  |
| 719. |         | $p\text{-AcNHC}_6\text{H}_4\text{CHO}$ , HI, m. $235^\circ$ . <sup>122</sup>     |
| 720. |         | Nicotinaldehyde, m. $110\text{--}16^\circ$ . <sup>177b</sup>                     |
| 721. |         | $\text{PhCH}_2\text{COCOOH}$ , m. $180^\circ$ . <sup>83</sup>                    |
| 722. |         | 1,2-Dihydro-1-methyl-2-pyrididyl CHO,<br>HI, m. $167^\circ$ dec. <sup>372</sup>  |
| 723. | Ethyl,  | $\text{Me}_2\text{CO}$ , m. $55^\circ$ . <sup>24</sup>                           |
| 724. |         | $o\text{-O}_2\text{NC}_6\text{H}_4\text{CHO}$ , m. $170.5^\circ$ . <sup>55</sup> |

|  |   |
|--|---|
| 725.   | $p$ -Me <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> CHO, monoHCl, m. 232°;<br>diHCl, m. 220° dec. <sup>55</sup> |
| 726.   | $p$ -(1-Pyrrolidyl)C <sub>6</sub> H <sub>4</sub> CHO, HCl, m. 245°<br>dec. <sup>55</sup>                        |
| 727. Propyl,   | Me <sub>2</sub> CO, m. 27°; b <sub>7</sub> 121°. <sup>24</sup>  |
| 728. Butyl,  | Me <sub>2</sub> CO, b <sub>10</sub> 146°. <sup>24</sup>   |
| 729.   | PhCHO, HCl, m. 186°. <sup>55</sup>  |
| 730. Octyl,  | PhCHO, m. 176°. <sup>55</sup>   |
| 731. Cetyl,  | PhCHO, HCl, m. 163°. <sup>55</sup>  |
| 732. Benzyl,   | $p$ -AcNHC <sub>6</sub> H <sub>4</sub> CHO, HCl, m. 216°. <sup>122</sup>  |
| 733. Et <sub>2</sub> NCH <sub>2</sub> CH <sub>2</sub> -, | PhCHO, diHCl, m. 192°. <sup>55</sup>  |
| 734. Allyl,  | Me <sub>2</sub> CO, m. 51°. <sup>24</sup>   |

Of H<sub>2</sub>NN:C(SR)NHR<sub>1</sub>

|              | R                                       | R <sub>1</sub>                                    |  |
|--------------|---|---|--|
| 735. Methyl, | Methyl,                                 | PhCH <sub>2</sub> COCOOH, m. 154°. <sup>83c</sup> |  |
| 736. Ethyl,  | Phenyl,                                 | PhCHO, m. 78°. <sup>55</sup>                      |  |
| 737.         | $p$ -BuOC <sub>6</sub> H <sub>4</sub> , | PhCHO, m. 90°. <sup>55</sup>                      |  |

**Thiosemicarbazones in Order of Their Melting Points**

To enable an investigator to learn which thiosemicarbazones melt at a given temperature, the 737 compounds in the preceding list are arranged according to their melting points. Where a range is given, or where there are several different figures, the highest is taken. Unfortunately, here as elsewhere there are many discrepancies in the recorded data. It is desirable to look at melting points several degrees above and below the figure in hand. The unreliability of recorded data is illustrated by the figures for one thiosemicarbazone: 215-7, 219, 219-20, 221, 224-6, 226-8.

| <i>M.p.</i> ° | No. | <i>M.p.</i> ° | No. | <i>M.p.</i> ° | No. | <i>M.p.</i> ° | No. | <i>M.p.</i> ° | No. |
|---------------|-----|---------------|-----|---------------|-----|---------------|-----|---------------|-----|
| 24            | 36  | 72            | 400 | 85            | 306 | 95            | 333 | 102           | 496 |
| 35            | 389 | 73            | 283 | 86            | 386 | —             | 491 | 104           | 14  |
| 51            | 734 | 74            | 392 | 87            | 666 | 96            | 465 | —             | 286 |
| 53            | 4   | 75            | 663 | 88            | 331 | —             | 651 | —             | 707 |
| 55            | 19  | 78            | 736 | 90            | 737 | 96.5          | 6   | —             | 717 |
| —             | 456 | 79            | 5   | 90.5          | 323 | 97            | 292 | 105           | 97  |
| —             | 723 | 81            | 284 | 92            | 395 | —             | 492 | —             | 383 |
| 65            | 3   | 82            | 402 | —             | 400 | 100           | 665 | —             | 387 |
| 66            | 283 | 84            | 39  | 93.5          | 385 | 101           | 653 | 106           | 50  |
| 67            | 285 | 85            | 17  | 95            | 247 | 102           | 165 | —             | 282 |

| <i>M.p.</i> ° | No. | <i>M.p.</i> ° | No. | <i>M.p.</i> ° | No. | <i>M.p.</i> ° | No. | <i>M.p.</i> ° | No. |
|---------------|-----|---------------|-----|---------------|-----|---------------|-----|---------------|-----|
| 107           | 291 | 126           | 46  | 144           | 84  | 152           | 53  | 160           | 699 |
| —             | 304 | —             | 269 | —             | 429 | —             | 501 | 161           | 221 |
| —             | 666 | 127           | 388 | —             | 488 | —             | 594 | —             | 289 |
| —             | 711 | 128           | 334 | —             | 587 | —             | 649 | —             | 320 |
| 108           | 12  | —             | 601 | —             | 677 | —             | 698 | —             | 488 |
| —             | 17  | —             | 716 | 145           | 71  | 153           | 10  | —             | 614 |
| —             | 323 | 129           | 588 | —             | 100 | —             | 418 | —             | 667 |
| —             | 669 | 130           | 354 | —             | 112 | 154           | 81  | 162           | 350 |
| 109           | 7   | 131.5         | 358 | —             | 364 | —             | 342 | —             | 624 |
| —             | 130 | 132           | 270 | —             | 387 | —             | 471 | —             | 661 |
| —             | 392 | —             | 313 | —             | 411 | —             | 520 | 163           | 28  |
| 110           | 47  | —             | 648 | —             | 485 | —             | 635 | —             | 510 |
| —             | 385 | —             | 696 | —             | 591 | 154.5         | 735 | —             | 589 |
| 111           | 8   | —             | 712 | 146           | 1   | 155           | 2   | —             | 595 |
| 112.5         | 44  | 134           | 681 | —             | 287 | —             | 29  | —             | 689 |
| 114           | 48  | 135           | 45  | —             | 390 | —             | 328 | —             | 731 |
| —             | 650 | —             | 428 | —             | 406 | —             | 354 | 164           | 195 |
| 115           | 246 | —             | 688 | —             | 432 | —             | 452 | —             | 218 |
| —             | 396 | 136           | 247 | —             | 541 | 156           | 390 | —             | 435 |
| 116           | 323 | —             | 387 | —             | 704 | 157           | 41  | —             | 705 |
| —             | 630 | —             | 592 | —             | 728 | —             | 300 | —             | 709 |
| —             | 720 | —             | 702 | 147           | 15  | —             | 311 | 165           | 59  |
| 117           | 294 | 137           | 30  | —             | 51  | —             | 434 | —             | 70  |
| —             | 385 | —             | 356 | —             | 240 | —             | 604 | —             | 93  |
| —             | 457 | —             | 715 | —             | 358 | 158           | 11  | —             | 312 |
| —             | 601 | 138           | 106 | —             | 386 | —             | 27  | —             | 401 |
| 118           | 49  | —             | 288 | —             | 668 | —             | 30  | —             | 439 |
| —             | 109 | —             | 426 | —             | 710 | —             | 298 | —             | 481 |
| —             | 303 | —             | 544 | 148           | 22  | —             | 360 | —             | 669 |
| —             | 357 | 139           | 78  | —             | 244 | —             | 477 | —             | 670 |
| 119           | 323 | —             | 425 | —             | 301 | —             | 481 | 166           | 67  |
| —             | 489 | 140           | 358 | —             | 660 | —             | 671 | —             | 94  |
| 121           | 103 | 141           | 464 | 148.5         | 43  | 159           | 350 | —             | 151 |
| —             | 281 | —             | 629 | —             | 290 | —             | 437 | —             | 236 |
| —             | 297 | 142           | 60  | 149           | 397 | —             | 634 | —             | 365 |
| —             | 424 | —             | 263 | —             | 471 | 160           | 9   | —             | 370 |
| —             | 727 | —             | 433 | —             | 592 | —             | 52  | —             | 405 |
| 122           | 368 | —             | 662 | 150           | 327 | —             | 204 | —             | 540 |
| 123           | 247 | —             | 685 | —             | 436 | —             | 296 | —             | 656 |
| 125           | 409 | —             | 695 | 151           | 302 | —             | 393 | —             | 682 |
| —             | 613 | 143           | 404 | 152           | 13  | —             | 468 | —             | 706 |
| —             | 664 | —             | 427 | —             | 23  | —             | 612 | 166.5         | 369 |
| —             | 676 | 144           | 37  | —             | 41  | —             | 672 | 167           | 9   |

| <i>M.p.</i> ° | No. | <i>M.p.</i> ° | No. | <i>M.p.</i> ° | No. | <i>M.p.</i> ° | No. | <i>M.p.</i> ° | No. |
|---------------|-----|---------------|-----|---------------|-----|---------------|-----|---------------|-----|
| 167           | 20  | 174           | 310 | 180           | 470 | 186           | 359 | 192           | 38  |
| —             | 301 | —             | 351 | —             | 638 | —             | 430 | —             | 111 |
| —             | 722 | —             | 431 | —             | 652 | —             | 480 | —             | 363 |
| 168           | 353 | —             | 700 | —             | 721 | —             | 494 | —             | 413 |
| —             | 410 | 174.5         | 229 | 181           | 40  | —             | 729 | —             | 500 |
| 169           | 80  | 175           | 58  | —             | 113 | 187           | 21  | —             | 733 |
| —             | 241 | —             | 273 | —             | 126 | —             | 73  | 193           | 159 |
| —             | 242 | —             | 331 | —             | 299 | —             | 363 | —             | 506 |
| 170           | 27  | —             | 382 | —             | 308 | —             | 486 | —             | 622 |
| —             | 69  | —             | 482 | —             | 394 | —             | 497 | 194           | 187 |
| —             | 111 | 176           | 9   | —             | 398 | —             | 602 | —             | 460 |
| —             | 132 | —             | 21  | —             | 440 | —             | 659 | —             | 504 |
| —             | 161 | —             | 92  | —             | 636 | 188           | 39  | —             | 535 |
| —             | 238 | —             | 139 | 181.5         | 317 | —             | 77  | —             | 590 |
| —             | 307 | —             | 280 | 182           | 239 | —             | 366 | —             | 597 |
| —             | 362 | —             | 394 | —             | 462 | —             | 371 | —             | 632 |
| —             | 367 | —             | 512 | —             | 567 | —             | 373 | —             | 640 |
| —             | 672 | —             | 518 | —             | 678 | —             | 645 | 195           | 54  |
| 170.5         | 724 | —             | 618 | —             | 684 | 188.3         | 143 | —             | 72  |
| 171           | 104 | —             | 644 | 183           | 82  | 189           | 68  | —             | 118 |
| —             | 110 | —             | 730 | —             | 202 | —             | 72  | —             | 128 |
| —             | 278 | 176.5         | 621 | —             | 206 | —             | 642 | —             | 169 |
| —             | 403 | 177           | 75  | —             | 382 | 190           | 20  | —             | 279 |
| —             | 412 | —             | 326 | —             | 471 | —             | 138 | —             | 293 |
| —             | 423 | —             | 329 | —             | 490 | —             | 220 | —             | 327 |
| —             | 438 | —             | 516 | —             | 697 | —             | 261 | —             | 343 |
| —             | 603 | —             | 628 | 184           | 115 | —             | 407 | —             | 359 |
| —             | 656 | 178           | 39  | —             | 482 | —             | 433 | 196           | 160 |
| —             | 701 | —             | 202 | —             | 542 | —             | 473 | —             | 319 |
| 172           | 42  | —             | 327 | —             | 617 | —             | 567 | —             | 324 |
| —             | 62  | —             | 425 | —             | 655 | —             | 620 | —             | 372 |
| —             | 83  | —             | 513 | —             | 686 | —             | 657 | —             | 417 |
| —             | 86  | —             | 637 | 185           | 74  | 190.5         | 107 | —             | 598 |
| 172.5         | 608 | 179           | 98  | —             | 79  | 191           | 55  | —             | 600 |
| 173           | 87  | —             | 114 | —             | 95  | —             | 56  | —             | 718 |
| —             | 127 | —             | 237 | —             | 111 | —             | 79  | 196.5         | 186 |
| —             | 306 | —             | 316 | —             | 281 | —             | 95  | 197           | 422 |
| —             | 361 | —             | 343 | —             | 343 | —             | 309 | —             | 490 |
| —             | 419 | 180           | 159 | —             | 370 | —             | 371 | —             | 507 |
| —             | 458 | —             | 201 | —             | 442 | —             | 375 | —             | 620 |
| —             | 658 | —             | 399 | —             | 607 | —             | 378 | 197.6         | 693 |
| 174           | 30  | —             | 408 | —             | 694 | —             | 495 | 198           | 89  |
| —             | 61  | —             | 448 | 186           | 214 | —             | 631 | —             | 168 |

| <i>M.p.</i> ° | No. | <i>M.p.</i> ° | No. | <i>M.p.</i> ° | No. | <i>M.p.</i> ° | No. | <i>M.p.</i> ° | No. |
|---------------|-----|---------------|-----|---------------|-----|---------------|-----|---------------|-----|
| 198           | 363 | 203           | 615 | 210           | 305 | 216           | 228 | 223           | 451 |
| —             | 375 | 204           | 20  | —             | 536 | —             | 529 | 223.5         | 344 |
| —             | 502 | —             | 131 | 211           | 69  | —             | 564 | 224           | 102 |
| —             | 522 | —             | 507 | —             | 158 | —             | 732 | —             | 137 |
| —             | 605 | —             | 521 | —             | 190 | 217           | 154 | —             | 192 |
| —             | 708 | —             | 611 | —             | 445 | —             | 158 | —             | 259 |
| 199           | 249 | 205           | 24  | —             | 683 | —             | 212 | —             | 274 |
| —             | 355 | —             | 167 | —             | 690 | —             | 449 | —             | 453 |
| —             | 455 | —             | 389 | 212           | 57  | —             | 614 | —             | 454 |
| 200           | 16  | —             | 443 | —             | 91  | —             | 623 | —             | 467 |
| —             | 187 | —             | 478 | —             | 148 | 218           | 203 | —             | 523 |
| —             | 234 | —             | 536 | —             | 155 | —             | 476 | —             | 577 |
| —             | 386 | —             | 610 | —             | 197 | —             | 505 | —             | 626 |
| —             | 609 | 206           | 170 | —             | 219 | —             | 585 | —             | 691 |
| —             | 610 | —             | 337 | —             | 225 | —             | 647 | —             | 703 |
| —             | 639 | 207           | 76  | —             | 250 | 218.5         | 108 | 225           | 140 |
| —             | 680 | —             | 90  | —             | 414 | 219           | 523 | —             | 158 |
| —             | 713 | —             | 130 | —             | 498 | —             | 545 | —             | 210 |
| 200.5         | 692 | —             | 132 | —             | 584 | —             | 546 | —             | 347 |
| 201           | 18  | —             | 175 | 213           | 32  | —             | 547 | —             | 525 |
| —             | 63  | —             | 249 | —             | 34  | 220           | 37  | —             | 614 |
| —             | 224 | —             | 250 | —             | 152 | —             | 66  | —             | 633 |
| —             | 230 | —             | 575 | —             | 265 | —             | 124 | 226           | 198 |
| —             | 251 | —             | 714 | —             | 352 | —             | 166 | —             | 268 |
| —             | 337 | 208           | 105 | —             | 517 | —             | 193 | —             | 526 |
| —             | 384 | —             | 119 | —             | 533 | —             | 336 | —             | 565 |
| —             | 593 | —             | 149 | 214           | 185 | —             | 465 | —             | 599 |
| —             | 643 | —             | 159 | —             | 204 | —             | 571 | —             | 674 |
| —             | 654 | —             | 257 | —             | 262 | —             | 646 | 227           | 130 |
| 202           | 245 | —             | 377 | —             | 576 | —             | 725 | —             | 132 |
| —             | 260 | —             | 441 | 214.5         | 211 | 221           | 527 | —             | 174 |
| —             | 390 | —             | 534 | 215           | 156 | 222           | 9   | —             | 314 |
| —             | 421 | —             | 550 | —             | 172 | —             | 205 | —             | 330 |
| —             | 559 | 209           | 129 | —             | 180 | —             | 250 | —             | 444 |
| —             | 679 | —             | 177 | —             | 235 | —             | 315 | —             | 551 |
| 202.5         | 264 | —             | 332 | —             | 325 | —             | 415 | —             | 625 |
| 203           | 65  | —             | 391 | —             | 474 | —             | 420 | 228           | 33  |
| —             | 85  | —             | 493 | —             | 487 | —             | 619 | —             | 64  |
| —             | 150 | —             | 519 | 216           | 96  | —             | 673 | —             | 143 |
| —             | 162 | 210           | 101 | —             | 117 | 223           | 123 | —             | 248 |
| —             | 188 | —             | 123 | —             | 136 | —             | 255 | —             | 463 |
| —             | 338 | —             | 147 | —             | 145 | —             | 340 | —             | 466 |
| —             | 606 | —             | 216 | —             | 146 | —             | 376 | —             | 534 |

| <i>M.p.</i> ° | No. | <i>M.p.</i> ° | No. | <i>M.p.</i> ° | No.   | <i>M.p.</i> ° | No. | <i>M.p.</i> ° | No. |
|---------------|-----|---------------|-----|---------------|-------|---------------|-----|---------------|-----|
| 229           | 120 | 233           | 503 | 240           | 129   | 247           | 475 | 262           | 557 |
| —             | 511 | —             | 578 | —             | 135   | —             | 553 | 263           | 450 |
| —             | 577 | 234           | 102 | —             | 144   | —             | 675 | —             | 524 |
| 229.5         | 227 | —             | 132 | —             | 266   | 248           | 619 | —             | 581 |
| 230           | 153 | —             | 134 | —             | 277   | 250           | 207 | 264           | 213 |
| —             | 173 | —             | 182 | —             | 340   | —             | 208 | —             | 379 |
| —             | 195 | —             | 252 | —             | 346   | —             | 223 | —             | 579 |
| —             | 196 | —             | 254 | —             | 374   | —             | 374 | 265           | 243 |
| —             | 199 | —             | 318 | —             | 416   | —             | 483 | —             | 381 |
| —             | 200 | —             | 459 | —             | 562   | —             | 509 | —             | 514 |
| —             | 209 | —             | 531 | —             | 574   | —             | 532 | —             | 548 |
| —             | 321 | 235           | 29  | 240.5         | 537.5 | —             | 687 | —             | 586 |
| —             | 335 | —             | 35  | 241           | 232   | 251           | 479 | 266           | 196 |
| —             | 339 | —             | 122 | —             | 566   | 252           | 133 | —             | 215 |
| —             | 523 | —             | 174 | 242           | 128   | —             | 213 | 267           | 128 |
| —             | 536 | —             | 194 | —             | 258   | —             | 561 | 268           | 515 |
| —             | 560 | —             | 217 | —             | 530   | 253           | 446 | 270           | 121 |
| —             | 578 | —             | 253 | —             | 539   | —             | 580 | 275           | 272 |
| 231           | 88  | —             | 267 | 243           | 199   | 255           | 130 | 276           | 164 |
| —             | 130 | —             | 549 | —             | 256   | —             | 483 | —             | 582 |
| —             | 469 | —             | 552 | —             | 619   | 256           | 194 | 280           | 142 |
| —             | 619 | —             | 554 | 244           | 314   | —             | 349 | —             | 181 |
| —             | 641 | —             | 596 | —             | 499   | 257           | 26  | —             | 183 |
| 232           | 99  | —             | 719 | 245           | 116   | —             | 233 | —             | 271 |
| —             | 226 | 236           | 176 | —             | 171   | —             | 276 | —             | 380 |
| —             | 315 | —             | 345 | —             | 344   | 258           | 231 | —             | 543 |
| —             | 379 | 237           | 31  | —             | 508   | —             | 341 | —             | 570 |
| —             | 514 | —             | 190 | —             | 572   | —             | 447 | 282           | 189 |
| —             | 528 | —             | 339 | —             | 573   | 259           | 121 | 283           | 610 |
| 233           | 154 | —             | 461 | —             | 726   | —             | 275 | 289           | 583 |
| —             | 178 | —             | 472 | 246           | 125   | 260           | 191 | 300           | 25  |
| —             | 219 | —             | 563 | —             | 184   | —             | 558 | —             | 627 |
| —             | 222 | 239           | 538 | 247           | 132   | —             | 568 | 320           | 163 |
| —             | 225 | —             | 555 | —             | 179   | 262           | 130 | 350           | 537 |
| —             | 344 | —             | 616 | —             | 340   | —             | 141 | 360           | 569 |
| —             | 484 | 240           | 35  | —             | 348   | —             | 322 | 380           | 556 |

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## CHAPTER 3

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# Dithizone

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### Introduction

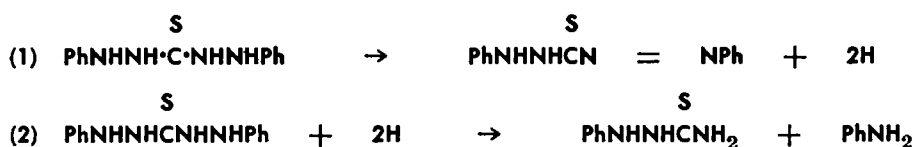
Dithizone,  $\text{PhNH}\cdot\text{NH}\cdot\text{CS}\cdot\text{N}:\text{NPh}$ , the phenyl hydrazide of phenylazothionoformic acid, also called, 1,5-diphenylthiocarbazone, may be considered to be a mixture of the thioketo and thioenol forms.<sup>310</sup> Influence of the solvent on the equilibrium has been studied.<sup>312</sup> Dithizone, designated Dz,<sup>186b</sup> is the most important member of a group of sulfur-nitrogen compounds that give, with heavy metal ions, colored salts or complexes which are extractable with organic solvents and are useful for the detection and determination of minute quantities of the metals.<sup>264</sup> Copper forms two complexes: (1) the normal reddish compound in alkaline media, corresponding to the cuprous dithizonate, and (2) an anomalous yellow compound in acidic media, containing two times more copper than the former. Mercury also forms two complexes, one pink and the other yellow.<sup>407</sup> The colored compounds are commonly formulated as salts but may be regarded as chelate or coordinate compounds.<sup>7, 82</sup> In this chapter they will be designated as *complexes*.

### Preparation

Dithizone was first prepared by Emil Fischer<sup>134</sup> in 1878, and later by Bamberger and coworkers,<sup>29</sup> by a method involving the diazo reaction which has been used for making the  $\alpha$ - and  $\beta$ -naphthyl- and other analogs.<sup>192, 314</sup> Directions for preparing dithizone



by Fischer's method have been given,<sup>175, 269, 275b</sup> detailed in *Organic Syntheses*.<sup>43</sup> To a solution of phenylhydrazine in benzene or ether, carbon disulfide is added. The yield of the salt,  $\text{PhNH}\cdot\text{NH}\cdot\text{CS}\cdot\text{SNH}_2\text{NPh}$ , is 96–8 per cent. When heated at  $98^\circ$ , this loses hydrogen sulfide and gives *s*-diphenylthiocarbohydrazide, which is refluxed for exactly five minutes in methanol containing potassium hydroxide. Hydrogen migrates from one molecule to another.<sup>207</sup>



Hydrogenation of dithizone with nickel catalyst gives two molecules of aniline.<sup>167</sup>

A number of homologs and analogs of dithizone have been prepared in the hope of obtaining more useful products.<sup>105, 303, 313, 323, 397, 398, 400</sup> The corresponding  $\beta$ -naphthyl compound has been prepared<sup>192, 388</sup> and used in a similar way for the estimation of traces of heavy metals,<sup>419</sup> particularly in biological materials.<sup>74, 75, 191c, 191d, 191e</sup> The ditolyl homolog has been proposed.<sup>389, 419</sup>

### Properties

Dithizone forms blue-black crystals soluble in carbon tetrachloride with green color, in alkalis with red, and in sulfuric acid with blue. Formulated as HDz it is a monobasic acid with an ionization constant of  $5.6 \times 10^{-6}$ ; <sup>201</sup> also  $2 \times 10^{-9}$ .<sup>23</sup> The molar absorptivity of dithizone in chloroform is  $40.6 \pm 0.5 \times 10^3$ .<sup>274a</sup> The molecular coefficient of extraction in the same solvent has been determined.<sup>247b</sup> Measurement of the distribution coefficient of dithizone between cyclohexane, carbon tetrachloride, benzene, and chloroform and aqueous buffer solutions of known *pH* lead to the values  $pK_{\text{HDz}} = 4.5 \pm 0.3$ .<sup>198</sup> Absorption spectra studies of dithizone have been made in a number of solvents and their values given.<sup>51, 87, 169, 318, 354</sup> Its primary dissociation constant has been calculated to be  $10^{-4.6 \pm 0.02}$ ; <sup>112</sup> also  $1.5 \times 10^{-5.339d}$ . The light absorption curve of 1,5- $\beta$ -naphthylthiocarbazone in carbon tetrachloride solution had two maximums, at  $665 \mu$  and at  $465 \mu$ . The molecular extinction coefficient was 40,000 at  $665 \mu$ . The dissociation constant was determined to be  $2.1 \times$

10<sup>-13</sup>. A comparison of calculated and experimental data shows it to be a monobasic acid.<sup>176</sup>

*G* values for gamma rays were determined for Dz in air-free and aerated carbon tetrachloride, chloroform, and mixtures of these with alcohol. Post-irradiation decomposition occurred in chloroform and in the alcohol mixtures.<sup>432</sup>

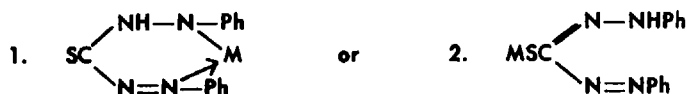
## Reactions

### ALKYLATION

Alkylation of dithizone takes place on the sulfur atom.<sup>198</sup> The methylation of dithizone gives two products, one violet and one yellow, and since in both of them the methyl is attached to the sulfur they must be stereoisomers; the yellow is considered to be *cis-cis* and the violet the *trans-trans* form.<sup>311</sup>

### METAL COMPLEXES

Hellmuth Fischer found that dithizone gives characteristic colored precipitates with heavy metal ions. He and his coworkers published more than seventy articles up to 1937.<sup>1b, 208</sup>



In this M represents one equivalent of a heavy metal. Structure 1 has been given<sup>440</sup> but 2 seems more probable on account of the known affinity of metals for sulfur. In 2, the alternation of double and single bonds is continuous. Most of these heavy metal salts are soluble in organic solvents such as carbon disulfide, chloroform, and carbon tetrachloride. Some of them are suitable for gravimetric, others for colorimetric determinations.

Metal "keto" dithizonates are shown to be derived by the replacement of the hydrogen of a mercapto rather than an imido group in diphenylthiocarbazone. "Keto" silver dithizonate in chloroform with methyl iodide gives a nearly black mono-methyl dithizonate. Since the latter is insoluble in alkali and does not form metal complexes, the acidic and metal-complexing properties of dithizone in forming "keto" derivatives must be due to the mercapto group.<sup>198</sup> A study of the free energies of formation of the dithizone complexes of copper and mercury has shown that

in the case of mercury, coordination appears to take place through the sulfur atom rather than through the nitrogen atoms, as in the case of nickel.<sup>107</sup> Copper is also anomalous.<sup>107</sup> Dissociation constants for the dithizone complexes of thallium, indium, nickel, cobalt, tin (II), bismuth, copper (II), silver, and mercury (II) have been given.<sup>317, 404</sup>

The structure of chelate complexes has been deduced from the structure of unimolecular films.<sup>133</sup> Structural formulas have been proposed for the cuprous and cupric dithizonate.<sup>319</sup>

Optical density measurements on the carbon tetrachloride extract of solutions of known lead and dithizone concentrations indicate that the constitution of the lead salt is  $\text{PbDz}_2$ .<sup>86</sup> The crystal structure of the mercury-dithizone complex has been found to be orthorhombic.<sup>180</sup>

Equilibria systems in the formation of dithizone-metal complexes have been investigated.<sup>51, 58, 161, 203, 204, 268</sup> Dithizone gives delayed reactions with mercuric, cobaltous, nickel, silver, and lead ions at  $-75^\circ$  to  $-100^\circ$  but it reacts almost instantaneously with ferrous ions.<sup>44</sup> The formation of complexes can be reversed by the addition of mineral acids, or of BAL, which form more stable complexes.<sup>202</sup> Reversion has been used in the determination of lead.<sup>200</sup>

By means of radioactive zinc chloride the exchange reactions between zinc dithizonate and zinc, copper, and cobalt ions were studied.<sup>261</sup> Other exchange reactions were investigated.<sup>157</sup> Dithizone is reported to be useful for the separation and purification of radium D, radium E, and polonium.<sup>47, 332.5</sup> The volatility of dithizone complexes has been studied; those of polonium and tellurium sublime readily.<sup>223, 259</sup> Dithizone has been used in the study of Liesegang-like rings<sup>131</sup> and of metal isotopes.<sup>5, 20, 359, 392</sup> It has reportedly played an important role in studying the mechanism of conduction in semi-conductors.<sup>404.5</sup> Dithizone reacts with dry nickel hydroxide and with nickel carbonyl in benzene solution.<sup>375a</sup>

### Dithizone in Analysis

Dithizone has been found to be useful in qualitative<sup>96, 103, 106, 165, 178, 275a</sup> and quantitative analysis, particularly where only traces of the heavy metals are present, as in vegetable and animal

products. Dithizone is more sensitive than hydroxyanthraquinone.<sup>254</sup>

A number of reviews on its applications have been written by Fischer<sup>135d, 135e, 135i, 137b, 137n, 139, 141</sup> and others.<sup>33, 69b, 101, 145, 228, 275e, 289, 317, 371, 374, 428a, 428g, 429, 435f, 440</sup> Recent developments have been reviewed by Iwantscheff.<sup>206</sup> Other chemists have supplied information on its use<sup>437</sup> and on making up the required solutions.<sup>81a, 83, 427b</sup> Its chloroform solution should be kept cold and in the dark.<sup>41</sup> A solution in carbon tetrachloride is stable almost indefinitely if stored in a cold, dark place under water containing sulfur dioxide.<sup>81d</sup>

Numerous articles have been written on analytical procedures.<sup>4, 11, 27, 31a, 37, 45, 52, 54, 73, 80, 81g, 96, 106, 126, 135b, 137c, 137h, 137i, 137j, 140, 142, 158, 159a, 165, 170, 177, 178, 179a, 181, 188, 193, 224, 237, 242, 263, 271, 275a, 316, 331, 336, 339c, 362, 395, 401, 410, 423, 433, 434, 438</sup> In general the heavy metal ions are taken out of their aqueous solutions by extraction with a solution of dithizone in chloroform or other suitable solvent.<sup>1a, 2, 9, 24, 65, 67, 94, 100, 111, 130, 137c, 160, 182, 199, 214, 236, 249, 291, 306, 326, 412, 416</sup> A shaking machine is useful.<sup>92</sup> The extraction follows the laws of mass action and distribution.<sup>83, 234</sup> Different metals are taken out at different pH values.<sup>1a, 124, 171, 189, 213, 227, 236, 249, 255b, 265, 292, 301, 304, 320, 328, 340, 354, 373, 382, 416, 439</sup> Copper, mercury (I and II), silver, gold, and palladium combine with dithizone in mineral acid solution. On the other hand, metals such as cobalt, nickel, lead, and zinc do not react appreciably with dithizone in acid solution but do combine completely in faintly ammoniacal solution.<sup>306</sup> Extraction time is greatly reduced in determining copper by employing either an ammoniacal or acetone solution.<sup>97</sup> Cadmium, zinc, tin, lead, and bismuth may be separated from mercury, copper, cobalt, and nickel by dilute hydrochloric acid.<sup>226</sup> Acetate and other buffers are used frequently.<sup>66, 95, 135j, 137a, 137e, 183, 330, 339a, 339b, 350a</sup> Bismuth can be removed from the dithizone-chloroform solution by washing it with acidified potassium iodide solution.<sup>171</sup> In the presence of potassium iodide, mercury is kept in the aqueous solution while copper is extracted.<sup>435a</sup> After the formation of the red lead-dithizone complex, the excess of the reagent can be taken out by washing with potassium cyanide solution.<sup>137c</sup> The separation and determination of microgram quantities of silver, mercury, and copper are possible.<sup>146</sup> A number of variations of the dithizone

method have been developed for mercury,<sup>59, 197, 217, 277, 411</sup> zinc,<sup>15, 66, 79, 93, 216, 280, 296, 442</sup> and lead.<sup>48, 115, 121, 162, 232, 239, 270, 274, 278, 365, 366</sup> Diethyl and diphenyl mercury may be estimated by a special procedure.<sup>276</sup>

The metal is usually estimated colorimetrically,<sup>8, 12, 18, 35, 42, 69a, 81c, 81e, 81f, 109, 118, 125, 126, 135a, 135c, 135f, 135j, 136, 137a, 137c, 137e, 137f, 137h, 137m, 137o, 142, 149, 155, 158, 210, 230, 260, 272, 290, 300, 315b, 327b, 334, 337, 342, 352, 353, 403, 406, 417, 428b, 430, 441</sup> photometrically,<sup>26a, 53, 54, 89, 159b, 177, 191a, 191b, 191c, 279, 292, 341, 384, 385, 386, 435a, 435e</sup> or polarographically.<sup>73, 182, 416, 441</sup> The "mixed-color" method is recommended in certain cases.<sup>38, 45, 102a, 137f, 137g, 137i, 174, 229, 237, 240b, 246, 265, 327b, 345, 406, 433</sup> The absorption spectra of dithizone, its oxidation products, and its copper and lead compounds have been studied.<sup>240a, 254, 290, 383</sup> Absorption curves are given for dithizone complexes of copper, silver, zinc, cadmium, mercury, thallium, lead, bismuth, cobalt, nickel, and tin.<sup>70, 141</sup> The dithizone complexes of practically all of the heavy metals can be used advantageously in producing spectra with electric sparks.<sup>333</sup> Traces of the metals may be extracted for this purpose.<sup>166</sup> Spectroscopic data for the estimation of cations in dithizone complexes in various solvents have been given.<sup>64, 202, 282, 399, 405</sup> The chromatographic separation of dithizone-metal complexes has been studied.<sup>17, 117, 370, 414</sup> Radiometric titration of nickel and lead against dithizone in presence of  $(\text{Co}^{60})\text{Cl}_2$  has been proposed.<sup>108</sup>

For the qualitative detection of heavy metals spot tests may be used.<sup>122, 123, 132, 135h, 309</sup> A dry, water-permeable acetyl-cellulose film impregnated with dithizone is convenient for such tests.<sup>376</sup>

Hydrogen sulfide and mercaptans may be titrated with mercuric or silver solutions, using dithizone as indicator.<sup>14, 244</sup>

As the solubility of the metal-dithizone complexes is small in water and great in chloroform and in carbon tetrachloride, small amounts of the heavy metals can be recovered from large volumes of water, as in the analysis of drinking water for heavy metals. The metals determined in this way are lead,<sup>102a, 130, 245, 292, 325, 328, 402a, 406</sup> copper,<sup>57, 328, 385, 386, 418</sup> zinc,<sup>8, 57, 114, 149, 328, 385, 386, 418</sup> bismuth, cadmium,<sup>328</sup> and silver.<sup>184, 235</sup> Mercury may be estimated in sea water.<sup>31a</sup> The corrosion of zinc by hot water has been studied by this means.<sup>173</sup> Certain enzymes interfere with the ability of dithizone to remove zinc.<sup>168</sup> The disadvantage of

the use of dithizone for determining trialkyl tin has been noted.<sup>321</sup> Other characteristics of the di- and trialkyl tin compounds have been mentioned.<sup>3</sup> The colorimetric determination of cadmium in effluents has been proposed for waste-disposal problems.<sup>357</sup> Methods for the determination of copper<sup>55</sup> and zinc<sup>356</sup> in nickel electroplating baths have been developed.

For the detection and estimation of traces of heavy metals in biological materials,<sup>258</sup> dithizone has given good service. In a comparison of methods for removing trace metals from microbiological media, treatment with dithizone, plus oxime, gave good results.<sup>104</sup> No definite connection has been found between complex formation and antibacterial action.<sup>347</sup> Traces of lead<sup>27, 28, 35, 69a, 72, 73, 127, 128, 129b, 185, 191a, 205, 240b, 293, 327c, 334, 346, 349, 350b, 351, 372, 402b, 403, 444</sup> with special reference to urine,<sup>35, 40, 102b, 130, 140, 215, 218, 219, 237, 326, 422</sup> to blood,<sup>27, 35, 49, 241, 267, 308, 338, 372, 396, 403, 422, 431</sup> and to bones;<sup>187, 257</sup> zinc<sup>8, 30, 129b, 349, 350b, 351</sup> in urine,<sup>31c, 95</sup> in blood,<sup>408, 413, 415</sup> and in human tissue;<sup>30, 163, 408</sup> mercury<sup>172, 196, 249, 265</sup> in urine;<sup>31b, 255a, 327b, 445</sup> bismuth also in urine<sup>191b, 327a</sup> and blood;<sup>191b</sup> and copper<sup>285, 349, 350b, 351</sup> as well as other metals<sup>77, 129b, 228, 266</sup> have all been determined with great precision<sup>46, 371</sup> in biological materials. Procedures for detecting<sup>156, 213, 215, 327b, 396</sup> and diagnosing<sup>338</sup> metal poisons have also been developed.<sup>156</sup>

Dithizone has been used to determine lead in food stuffs,<sup>49, 142, 205, 231, 283, 351, 385, 386, 428c</sup> in maple and sugar products,<sup>81e, 81f, 170, 315a, 315b, 332, 433</sup> on sprayed fruit,<sup>81b, 369, 427a, 428b, 430, 436</sup> in milk,<sup>25, 129a, 155</sup> and in canned foods;<sup>69b, 238, 243, 252</sup> also for determining zinc in foodstuffs;<sup>6, 49, 67, 114, 186a, 188, 221, 281, 316, 331, 351, 358, 368, 385, 386, 394, 401, 428c, 428f</sup> copper in food products,<sup>36, 49, 80, 171, 361, 385, 386, 395, 428c, 428f</sup> specifically in milk,<sup>61, 286, 352, 353, 395</sup> canned food,<sup>252, 295</sup> and on apples;<sup>335</sup> mercury in sprayed fruit<sup>16, 144, 225</sup> and wheat,<sup>12</sup> and in other foods;<sup>249, 428c, 435b, 435c, 435d, 435e</sup> cadmium in food products;<sup>225, 252, 363</sup> and tin in beer.<sup>80</sup> In addition certain other proposed procedures for metals in foods<sup>38, 428d, 428e</sup> use dithizones. The dithizone method is well adapted to the estimation of the infinitesimal amounts of the heavy metals which seem to be necessary constituents of plants<sup>49, 68, 89, 90, 148, 150, 188, 222, 302, 307, 360, 380, 416, 425, 435a</sup> and must be present in fertile soils.<sup>49, 186c, 345, 361, 425</sup> Lead,<sup>220</sup> copper, and zinc<sup>159a</sup> can be satisfactorily determined in fertilizers. It has been employed in the detection of contamination by heavy metals in medicinals,<sup>26b, 34, 39, 45, 127,</sup>

212, 273, 287, 377, 378, 384 cosmetics,<sup>60, 348</sup> dyes,<sup>9</sup> and oils.<sup>19, 164, 294, 324</sup> Dithizone detects 0.0004% of zinc, 0.001% of copper, and 0.003% of lead in essential oils.<sup>147</sup> Lead<sup>181, 322, 420</sup> and mercury<sup>246</sup> in air, and copper, zinc, and lead<sup>288</sup> in dust and smoke have been estimated. Lead can be determined in gasoline,<sup>364, 375b</sup> in sulfuric acid,<sup>91</sup> and in earthenware glazes.<sup>426</sup>

The dithizone method is suitable for estimating a small amount of one metal present as an impurity in another, such as lead in iron,<sup>305</sup> steel,<sup>38</sup> magnesium,<sup>379</sup> or aluminum;<sup>85</sup> zinc in steel,<sup>54</sup> lead,<sup>84</sup> tin,<sup>233</sup> or aluminum;<sup>85, 137o</sup> bismuth in lead or copper;<sup>13a, 13b, 179a</sup> cobalt to 0.1% in nickel;<sup>135g</sup> copper in zinc<sup>116, 137i</sup> or nickel;<sup>230</sup> and cadmium in zinc<sup>137i, 137j</sup> down to one part in 10,000.<sup>137m</sup> Lead down to 0.05% can be found in zinc, cadmium, copper, mercury, silver, arsenic, tin, aluminum, and bismuth.<sup>137a</sup> It has been determined in copper, nickel, and cobalt.<sup>443</sup> Mercury can be estimated in zinc, lead, cadmium, and bismuth down to 0.0001%; in copper, to 0.001%; and in silver, to 0.1%.<sup>137k</sup> The trace impurities in a specimen of metal may indicate its origin.<sup>297</sup> From 0.2 to 0.005 mg. of thallium can be determined in the presence of other metals.<sup>179b</sup> Silver has been detected down to 0.1 gamma,<sup>138, 344</sup> mercury to 1.5 gamma,<sup>410</sup> indium to 1–2 gamma,<sup>410</sup> and zinc down to 0.02.<sup>96, 423</sup> 0.005 to 0.1 mg. of bismuth can be identified.<sup>179a</sup> Dithizone in molten naphthalene is said to detect 0.004 gammas of bismuth by spot test procedure.<sup>50, 63</sup>

In criminal investigations dithizone may aid in identifying lead, copper, and zinc in fabrics that have been penetrated by bullets.<sup>56, 119, 190</sup>

### Other Proposed Uses

Dithizone and some of its derivatives have been tested as to their usefulness in a number of fields: in rubber chemistry in vulcanization,<sup>251</sup> as an initiator of oxidation,<sup>298</sup> as a plasticizer,<sup>284</sup> and as an inhibitor of the oxidation of rubber solutions;<sup>299</sup> as a stabilizer for carotene,<sup>40</sup> ether,<sup>262</sup> and hydrazine;<sup>21</sup> also to inhibit the tarnishing tendency of detergents,<sup>393</sup> and to increase rot-resistance of cotton duck.<sup>194</sup> Chloroform, carbon tetrachloride, and ethyl alcohol solutions of dithizone have been investigated as potential dosimeters for ionizing radiations.<sup>78</sup> It has been used to detect traces of metals volatilized in steam,<sup>387</sup>

and also to determine the contamination of distilled water caused by contact with vulcanized rubber.<sup>76</sup>

Dithizone gives a color with milk that has been heated as high as 85°. It can show the presence of less than 5% of heated milk in raw.<sup>113</sup> It is a sensitive test for bromine<sup>10</sup> and can be used in the microdetermination of halogens.<sup>381</sup>

### Physiological

Dithizone produces diabetes in rabbits<sup>62, 99, 152, 153, 195, 211</sup> and in rats<sup>209</sup> but not in dogs.<sup>2, 88</sup> Dithizone at 50 mg./Kg. causes permanent diabetes in rabbits.<sup>211</sup> Cystine is said to give some protection.<sup>32</sup> Eye effects are also produced by dithizone which causes cataracts and lesions of the retina in rabbits.<sup>22, 195, 343</sup> Symptoms and biochemical changes produced by dithizone in animals have been described.<sup>98, 99, 143, 151, 154, 390, 391</sup> Some attention has been given to the protective action of dithizone in mercury poisoning<sup>421</sup> and in thallium poisoning in rats,<sup>256</sup> and to protection against radio-lesions.<sup>248</sup> There were no noteworthy results.

The activities of certain enzymes are inhibited by dithizone or its metal complexes.<sup>250, 400, 424</sup> It has no notable effect on staphylococci or pleuro-pneumonia organisms.<sup>253</sup>

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## CHAPTER 4

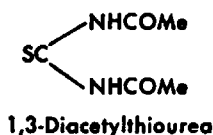
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# Thiobarbituric Acid

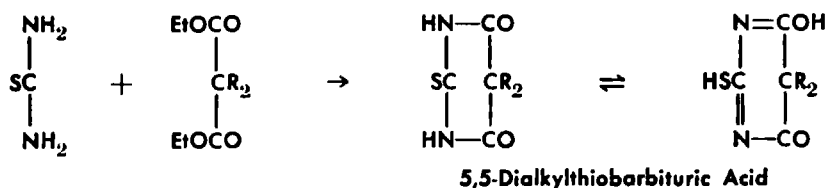
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### Introduction

Thiourea may be acylated in one or in both of the amino groups. Acylation by malonic acid should give the cyclic compound which is called thiobarbituric acid on account of its analogy to barbituric acid.



The formation of thiobarbituric acids is similar to that of the barbiturics; a malonic ester is condensed with thiourea. An alkyl malonic ester gives the corresponding alkylthiobarbituric acid:



There are tautomeric forms as is the case with the barbituric acids. A number of the 5,5-dialkylthiobarbituric acids have soporific and other valuable physiological properties. In the search for pharmaceuticals with enhanced efficiency and low toxicity, thiobarbituric acids containing all manner of substituents in all possible positions have been synthesized and studied.



Those with one, or preferably two, alkyls in the 5-position have received the most attention.

### Preparation

The general method of preparing these compounds is to add one molecule of a malonic ester and a moderate excess of thiourea to two of sodium ethylate in absolute alcohol. The mixture is refluxed to form the disodium salt. The free thiobarbituric acid separates on the addition of aqueous mineral acid. Much attention has been given to their preparation: 1, 2, 13, 42, 47, 52, 53, 54, 67, 68, 69, 79, 89, 114, 123, 135, 140, 142, 161, 165, 176, 178, 179, 180, 194, 219, 235, 246, 257.

Sodium metal, sodamide,<sup>168</sup> or magnesium methylate<sup>176</sup> may be employed as condensing agents. Lithium ethylate is said to give an excellent yield, 80% of the theoretical.<sup>101</sup>

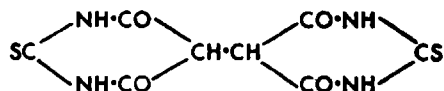
A number of thiobarbituric acids, having one of the groups,  $\text{RSCH}_2$ -,  $\text{RSCHR}'$ -,  $\text{RSCH}_2\text{CH}_2$ -, and  $\text{RSCH}_2\text{CHMe}$ -, as a substituent in the 5-position have been prepared.<sup>249, 263</sup> Thiobarbiturates of 1,2,3-trisubstituted pyrazolones may be prepared by treating an aldehyde of the pyrazolone with malonyl-thiourea.<sup>159, 169</sup> Spiro thiobarbituric acids have been synthesized by adding  $(\text{CH}=\text{CH}_2)_2$  to dialkyl alkylidenemalonates and condensing the ester formed with thiourea.<sup>53, 242</sup> A more involved method of preparation has been used to prepare the spiro derivatives which have sedative and hypnotic therapeutic value.<sup>165</sup> Certain aldehydes and thiopyrimidine derivatives yield thiobarbituric acids.<sup>206</sup>

Malononitrile and its alkyl derivatives have been condensed with thiourea and the resulting di-iminothiopyrimidines have been saponified to the corresponding thiobarbituric acids.<sup>85</sup> The  $\alpha$ -alkyl- $\alpha$ -carbethoxy- $\gamma$ -butyric lactones condense with thiourea in the presence of sodium ethylate to form the corresponding 5-alkyl-5-(2-hydroxyethyl)-2-thiobarbituric acids. Melting points of some of these compounds are given in the table of properties.<sup>224</sup>

Certain 5,5-disubstituted 1-methyl-2-thiobarbituric acids may be prepared by condensing a substituted cyanoacetic ester with N-methylthiourea to form the corresponding substituted imino-2-thiobarbituric acid, which is then hydrolyzed to the desired 1-methyl-2-thiobarbituric acid or, alternatively, by condensing

a diester of a substituted malonic acid with N-methylthiourea in the presence of sodium ethylate.<sup>66, 165</sup> A dialkyl malonyl chloride condenses with thiourea.<sup>176</sup>

From the ethyl ester of ethanetetra-carboxylic acid a double thiobarbituric acid has been made:<sup>209</sup>



A thiobarbituric acid can be prepared by treating a dialkyl-malonamide with carbon disulfide and sodium ethylate.<sup>85</sup>

S-Alkylisothioureas,  $\text{RSC}(:\text{NH})\text{NH}_2$ , condense with malonic esters to give 2-alkylthiobarbituric acids.<sup>76, 133, 139, 258</sup> These can be prepared, the other way around, by reacting the sodium salt of a thiobarbituric acid with an alkyl halide.<sup>161, 257</sup> The velocities with various alkyl iodides have been compared.<sup>189</sup>

N-Alkylthiobarbituric acids are prepared from N-alkylthioureas.<sup>58</sup> A few thiobarbituric acids having alkyls on one or both of the nitrogens in the 1 and 3 positions have been synthesized.<sup>18</sup>

2-Thiouramil, which is 5-aminothiobarbituric acid, is obtained from aminomalonic ester.<sup>141</sup>

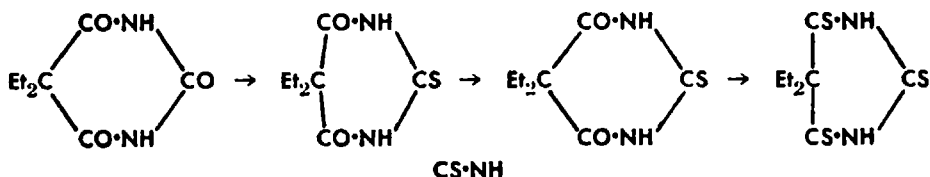
A 5,5-dialkyl-4-thiobarbituric acid can be obtained by the action of hydrogen sulfide on the corresponding 4-iminobarbituric acid. To prepare a 2,4-dithiobarbituric acid ethyl dialkyl cyanacetate,  $\text{R}_2\text{C}(\text{CN})\text{CO}_2\text{Et}$ , is condensed with thiourea to the 5,5-dialkyl-4-iminothiobarbituric acid, which is then treated with hydrogen sulfide.<sup>31</sup>

### Reactions of Thiobarbituric Acids

5-5-Dialkylthiobarbituric acids can be desulfurized to the corresponding dialkylbarbituric acids by heating with mineral acids, with or without an oxidising agent,<sup>77, 79</sup> or with a heavy metal salt.<sup>86</sup> A 4-imino-group is removed along with the sulfur.<sup>85</sup> Reductive desulfurization of thiobarbituric acids with Raney nickel has been carried out, the thiobarbituric acids going to the corresponding hydropyrimidines.<sup>228, 256</sup> Reduction with sodium amalgam gives a mixture which contains some of the barbiturate and some 5,5-diethyl-4,6-dioxy-2,5-dihydropyrimidine along with considerable 5,5-N,N'-diethylmalonamide.<sup>78, 79</sup>

Conversely, the oxygen atoms of 5,5-dialkylbarbituric acids

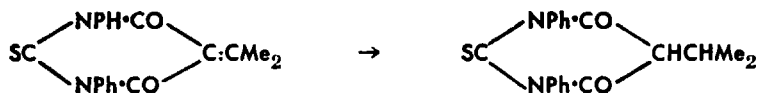
may be substituted, by sulfur, one after another, by heating them in a hydrocarbon solution with phosphorus tri- or pentasulfide.<sup>47, 122</sup>



With ammonia the di- and trithio- compounds give the corresponding 4-imino- derivatives.<sup>47</sup> The melting points of some di- and trithio- derivatives are in the tables of properties.

Equivalent amounts of sodium thiobarbiturate and sodium nitrate in aqueous solution give the sodium salt of thiovioluric acid.<sup>137</sup> Ethyl nitrite converts 1,3-diphenylthiobarbituric acid into diphenylthiovioluric acid.<sup>164, 259</sup> Isonithosothiobarbituric acid is prepared by passing oxides of nitrogen into a cold aqueous suspension of thiobarbituric acid.<sup>156</sup> The 4-iminothiobarbituric acids have been extensively studied and compared with the corresponding thiobarbituric acids.<sup>189</sup>

Thiobarbituric acid condenses with aldehydes.<sup>149</sup> The aldehyde and ketone derivatives of 1,3-diphenylthiobarbituric acids can be reduced to 5-alkyl-substituted acids:<sup>259</sup>



### Identification and Detection

Thiobarbituric acid derivatives give complex salts with the heavy metals.<sup>134, 144, 185</sup> The copper salts have been intensively studied.<sup>189</sup> This fact has been used in analysis.<sup>60</sup> The melting points and locations of maximum absorption bands have been given for the heavy metal salts of isonitrosodiphenyl-thiobarbituric acid.<sup>94</sup> The metal salts of the thiovioluric acids display a wide range of colors.<sup>137, 223</sup> 1,3-Diphenyl-2-thiobarbituric acid forms a highly colored, orange-red thallous complex with a thal- lous salt and carbon disulfide.<sup>197</sup>

Trithiobarbituric acid is a valuable reagent for the detection of heavy metals: silver ions, one in 20,000,000; cuprous, one in 5,000,000; cupric, one in 10,000,000; and mercuric, one in 2,000,000. *p*-Dimethylaminobenzylidenethiobarbituric acid is also a

sensitive reagent, particularly for gold and palladium.<sup>196</sup> Diphenylthiovioluric acid, a derivative of 5-isonitrosobarbituric acid, has been used as a reagent for the gravimetric estimation of copper.<sup>223</sup>

Various oxidised vegetable oils react with thiobarbituric acid to produce a characteristic red color.<sup>138</sup> Thiobarbituric acid also reacts with the ammonium salt of enolglutaconaldehyde formed from pyridine by ultraviolet irradiation; a lilac to bluish-green polymethine dye is produced which can serve for the detection of this acid.<sup>92</sup>

Good results are claimed for a color reaction for the detection of thiobarbiturates which depends on the formation of the green color of a cobalt complex.<sup>119, 270</sup> 5,5-Dialkylthiobarbituric acids can be determined volumetrically with NaOI;<sup>265</sup> also argentometrically.<sup>264</sup>

The separation and identification of barbiturates and thiobarbiturates in pure drugs by paper chromatography and micro-crystallography has been evaluated.<sup>12, 150, 260</sup> A comparison has been made of the polarographic determination with the volumetric determination of thiobarbituric acid derivatives.<sup>173</sup> The infrared<sup>155, 240, 267</sup> and ultraviolet absorption spectra<sup>171</sup> of thiobarbituric acid and derivatives have been investigated. Electro-metric methods have been studied for the determination of barbituric and thiobarbituric acids.<sup>8</sup>

### Application

Certain thiobarbiturates have been investigated for their value in anesthesia and analgesia, and as hypnotics. References to these are given in the section entitled *Pharmacological*. The literature also contains references to other applications.

Thiobarbituric acid has been used in testing for oxidation products of unsaturated fatty acids.<sup>64, 99, 261</sup> It is claimed that the so-called thiobarbituric acid (TBA) test provides a numerical measure of oxidative rancidity in oat cereals and other dry baked products.<sup>44</sup> It is said to be reliable as an index of rancidity in frozen pork<sup>239</sup> and as a measure of oxidation in many fat-containing foods;<sup>195, 210</sup> also in following oxidation in cottonseed and soy bean oils.<sup>220</sup> The test has been used for measurement of rancidity in fishery products.<sup>267</sup>

In the preservation of milk by radiation the so-called TBA

test is reported to be useful as a means of evaluating changes induced by radiation.<sup>195, 255</sup> The test is also said to give an indication of flavor and contamination by iron in dairy products.<sup>49, 74, 98, 236</sup> TBA reagent has been applied to a study of pathological tissues.<sup>65, 157</sup>

Oxonol<sup>132</sup> and merocyanine dyes<sup>243, 268</sup> have been synthesized, using thiobarbituric acid or its derivatives as the starting materials.<sup>268</sup> The merocyanine dyes have been studied in connection with photographic sensitizers and with absorption as an indication of stabilization.<sup>36</sup> Monomethine dyes containing a thiobarbituric acid nucleus are claimed to be useful in preparing backing layers for photographic plates and films.<sup>147</sup> Thiobarbituric acid derivatives have been investigated for suitability as retarders for photographic emulsions<sup>186, 187</sup> and as filters and antihalation dyes.<sup>148</sup> 2-Thiobarbituric acid has also been studied as an aid in forming azo dye images in photographic material.<sup>212</sup> Certain thiobarbituric acids form silver salts which cannot be reduced by conventional photographic color developers but are soluble in the fixing bath.<sup>212</sup>

### Pharmacological

The physiological effects of the thiobarbiturates have been extensively studied. There are reviews by Thomas,<sup>238</sup> and also by other investigators.<sup>9, 163, 247</sup> References to a number of articles are given without discussion. These references relate chiefly to anesthesia, analgesia, and hypnosis.<sup>10, 15, 16, 20, 21, 22, 23, 29, 32, 40, 48, 50, 53, 54, 59, 61, 66, 73, 81, 82, 83, 93, 95, 105, 106, 107, 109, 110, 112, 116, 122, 128, 129, 133, 136, 151, 152, 153, 160, 162, 165, 172, 174, 185, 188, 190, 192, 201, 203, 204, 211, 218, 221, 222, 226, 229, 231, 233, 234, 238, 242, 245, 247, 252, 253, 254, 262, 269</sup>

Short-acting thiobarbiturates<sup>30, 35, 66, 81, 90, 91, 111, 165, 177, 232</sup> have been investigated and also the effect that others have on the liver<sup>17, 19, 25</sup> and other organs.<sup>33, 46</sup>

Sodium pentothal is one of the most important thiobarbiturates in use as an anesthetic. Chemically it is sodium 5-ethyl-5-(1-methylbutyl)-2-thiobarbituric acid. Its application has gradually expanded and it is now widely used in hospitals and in private practice.<sup>7, 26, 113</sup>

The effect of various concentrations of pentothal and the amount necessary to produce anesthesia have been investigated both for man<sup>37, 118, 128, 191, 253</sup> and for animals.<sup>21, 41, 43, 62, 87, 96,</sup>

100, 103, 106, 115, 117, 121, 125, 126, 128, 131, 151, 167, 172, 198, 199, 202, 208, 226, 227, 244

Sodium pentothal has been studied in comparison with barbiturates and thiobarbiturates.<sup>24, 111, 136, 145, 192</sup> Radioactive sodium pentothal injected intravenously is rapidly distributed to all tissues of the body. Within thirty seconds most of it is out of the blood and found fairly evenly dispersed in the various organs.<sup>28, 237</sup> A comparison as to metabolism and distribution with other thiobarbiturates has been made.<sup>39, 119, 146, 252</sup> When pentothal was in the stage of early experimentation certain tests tended to show that it produced focal necrosis of the liver in mice and dogs.<sup>203, 204</sup> Later it was shown that pentothal was a safe anesthetic to employ under properly controlled conditions.<sup>57, 166, 214, 250</sup> Carbohydrate metabolism, which is directly related to hepatic functions, has been investigated;<sup>102, 130, 158, 230</sup> and also insulin shock during anesthesia.<sup>193</sup>

The respiratory effect of pentothal has been studied,<sup>70, 72, 105, 182, 183, 184, 200, 241</sup> as well as the cardiovascular effects.<sup>20, 56, 83, 107, 108, 110, 153, 245</sup> The physiology of shock has been described.<sup>80, 207</sup> Studies have been made on brain waves during pentothal anesthesia,<sup>34, 88, 97, 124, 127</sup> and also on the determination of oxygen and carbon dioxide in the blood during anesthesia.<sup>14, 84, 216</sup> In obstetrics, pentothal has shown favorable results.<sup>32, 71, 170</sup> Assay methods for sodium pentothal have been developed,<sup>251</sup> including its recovery after being added to serum.<sup>51, 5</sup>

Reviews have been written on its pharmacological action and range of usefulness<sup>9, 163, 238, 247</sup> and other reports in the literature attest to the value of pentothal as an anesthetic.<sup>190, 241</sup> In 1045 cases, 96.8% showed satisfactory results,<sup>95</sup> and in one hundred (ophthalmic) cases pentothal proved to be an ideal anesthetic.<sup>229</sup> Favorable reports were also made in 3,559 cases.<sup>16</sup>

### Physical Properties of Some Thiobarbituric Acids

#### 5-MONOSUBSTITUTED-2-THIOBARBITURIC ACIDS



R

Methyl, m. 244°.<sup>257</sup>

Ethyl, m. 192°,<sup>79</sup> 191°,<sup>257</sup> 174°.<sup>161</sup>

*i*-Propyl, m. 178°,<sup>168</sup> 173°.<sup>161</sup>

## R

- i*-Amyl, m. 162°. <sup>256</sup>  
*s*-Amyl, m. 148°. <sup>67</sup>  
 Anilinomethylene, m. 335° dec. <sup>268</sup>  
*p*-Methylanilinomethylene, m. 333° dec. <sup>268</sup>  
*m*-Hydroxyanilinomethylene, m. 326° dec. <sup>268</sup>  
*p*-Methoxyanilinomethylene, m. 327° dec. <sup>268</sup>  
 Acetanilinomethylene, m. 337° dec. <sup>268</sup>  
*m*-Acetoxyacetanilinomethylene, 267° dec. <sup>268</sup>  
 β-Hydroxy-α-naphthylmethylene, m. 265° dec. <sup>268</sup>  
 1-Cyclohexenyl, m. 228°. <sup>2</sup>  
 2-Cyclohexenyl, m. 155°. <sup>2</sup>  
 2-Cyclo-octenyl, m. 246°. <sup>143</sup>  
 Phenyl, m. 257°, <sup>256</sup> 254°. <sup>75</sup>  
 Benzyl, m. 220°. <sup>246</sup>

## 5,5-DISUBSTITUTED-2-THIOBARBITURIC ACIDS



## R      R'

- Methyl, Methyl, m. 216°. <sup>79</sup>  
     2-Nitrobutyl, m. 203° dec. <sup>11</sup>  
     *i*-Butyl, m. 120°. <sup>133</sup>  
     Cyclo-octyl, m. 167°. <sup>143</sup>  
     *i*-Propenyl, m. 155°. <sup>54</sup>  
     2-Methallyl, m. 182°, <sup>235</sup> 131°. <sup>246</sup>  
     1-Heptenyl, oil. <sup>114</sup>  
     1-Cyclohexenyl, m. 173°. <sup>75</sup>  
     Phenyl, m. 213°. <sup>75</sup>  
 Ethyl, Ethyl, m. 180°, <sup>89</sup> 177°, <sup>13, 93</sup> 174.5°. <sup>179</sup>  
     Propyl, m. 174.5°. <sup>179</sup>  
     *i*-Propyl, m. 192°. <sup>235, 246</sup>  
     Butyl, m. 145°. <sup>235, 246</sup>  
     2-Nitrobutyl, m. 152° dec. <sup>11</sup>  
     *i*-Butyl, m. 173°, <sup>133</sup> 170°. <sup>179</sup>  
     *s*-Butyl, m. 165°. <sup>235, 246</sup>  
     1-Methyl-3-carboxypropyl, m. 140°. <sup>266</sup>  
     *i*-Amyl, m. 181°, <sup>5</sup> 177°, <sup>246</sup> 176°, <sup>13</sup> 169°, <sup>235</sup> 143°. <sup>154</sup>  
     *s*-Amyl, m. 159°, <sup>235, 246</sup> 157°. <sup>3</sup>  
     Hexyl, m. 137°. <sup>235</sup>

| R                 | R'   |
|-------------------|--|
|                   | 1-Methylbutyl, m. $94^{\circ}$ ; b <sub>1.5</sub> $140^{\circ}$ . <sup>249</sup>                             |
|                   | 1-Ethylbutyl, m. $165^{\circ}$ . <sup>154</sup>  |
|                   | 2-Ethylbutyl, m. $138^{\circ}$ . <sup>235, 246</sup>   |
|                   | Cyclopentyl, m. $173^{\circ}$ . <sup>246</sup>   |
|                   | Cyclohexyl, m. $207^{\circ}$ . <sup>235, 246</sup>   |
|                   | 2-Bromocyclohexyl, m. $193^{\circ}$ . <sup>52</sup>  |
|                   | Ethoxymethyl, m. $147^{\circ}$ . <sup>42</sup>   |
|                   | 2-Hydroxyethyl, m. $163^{\circ}$ . <sup>224</sup>  |
|                   | Ethylmercaptomethyl, m. $138^{\circ}$ . <sup>27</sup>  |
|                   | Ethylsulfonylmethyl, m. $225^{\circ}$ . <sup>27</sup>  |
|                   | Allyl, m. $173^{\circ}$ . <sup>235, 246</sup>  |
|                   | <i>i</i> -Propenyl, m. $192^{\circ}$ , <sup>54</sup> $191^{\circ}$ . <sup>53</sup>                           |
|                   | 1-Butenyl, m. $122^{\circ}$ . <sup>114</sup>   |
|                   | 3-Chlorobutenyl, m. $130^{\circ}$ . <sup>235, 246</sup>  |
|                   | 2-Methallyl, m. $161^{\circ}$ . <sup>235, 246</sup>  |
|                   | <i>i</i> -Butenyl, m. $129^{\circ}$ . <sup>114</sup>   |
|                   | 1-Methyl-1-butenyl, m. $152^{\circ}$ . <sup>55</sup>   |
|                   | 1-Methyl-3-butenyl, m. $156^{\circ}$ . <sup>266</sup>  |
|                   | 1-Methyl-4-pentenyl, m. $140^{\circ}$ . <sup>266</sup>   |
|                   | 1-Propyl-1-butenyl, m. $169^{\circ}$ . <sup>53</sup>   |
|                   | 2-Cyclopentenyl, m. $195^{\circ}$ . <sup>48</sup>  |
|                   | 1-Cyclohexenyl, m. $189^{\circ}$ , <sup>2</sup> $188^{\circ}$ , <sup>246</sup> $186^{\circ}$ . <sup>75</sup> |
|                   | 3-Methylcyclohexenyl, m. $155^{\circ}$ . <sup>246</sup>  |
|                   | Phenyl, m. $220^{\circ}$ , <sup>75</sup> $217^{\circ}$ , <sup>235, 246</sup> $212^{\circ}$ . <sup>248</sup>  |
|                   | Benzyl, m. $260^{\circ}$ . <sup>175</sup>  |
| Propyl,           | Propyl, m. $154^{\circ}$ . <sup>79</sup>   |
|                   | <i>i</i> -Propyl, m. $168.5^{\circ}$ . <sup>179</sup>  |
|                   | Butyl, m. $135.5^{\circ}$ . <sup>179</sup>   |
|                   | <i>i</i> -Butyl, m. $132^{\circ}$ . <sup>179</sup>   |
|                   | <i>s</i> -Butyl, m. $165^{\circ}$ . <sup>179</sup>   |
|                   | Hexyl, m. $114.4^{\circ}$ . <sup>179</sup>   |
|                   | 2-Hydroxyethyl, m. $151^{\circ}$ . <sup>224</sup>  |
|                   | Allyl, m. $138^{\circ}$ . <sup>179</sup>   |
|                   | Propenyl, m. $151^{\circ}$ . <sup>55</sup>   |
|                   | <i>i</i> -Propenyl, m. $185^{\circ}$ . <sup>54</sup>   |
|                   | Methallyl, m. $158^{\circ}$ . <sup>67</sup>  |
|                   | Cyclopentenyl, m. $127^{\circ}$ . <sup>48</sup>  |
| <i>i</i> -Propyl, | Butyl, m. $153^{\circ}$ . <sup>175</sup>   |
|                   | <i>i</i> -Butyl, m. $117^{\circ}$ . <sup>179</sup>   |



| R                                   | R'   |
|-------------------------------------|--|
|                                     | Amyl, m. 98.5°. <sup>179</sup>                         |
|                                     | Cyclohexyl, m. 187°. <sup>246</sup>                    |
|                                     | Butylmercaptoethyl, m. 70°. <sup>263</sup>             |
|                                     | Allyl, m. 180°, <sup>68</sup> 176.5°. <sup>179</sup>   |
|                                     | Propenyl, m. 141°. <sup>55</sup>                       |
|                                     | 1-Butenyl, m. 110°, <sup>114</sup> 108°. <sup>55</sup> |
| Butyl, <i>i</i> -Butyl,             | m. 111°. <sup>133</sup>                                |
|                                     | Propenyl, m. 128.5°. <sup>55</sup>                     |
|                                     | Allyl, m. 121°. <sup>179</sup>                         |
|                                     | <i>i</i> -Propenyl, m. 161°. <sup>54</sup>             |
|                                     | Methallyl, m. 137.5°. <sup>67</sup>                    |
|                                     | 2-Hydroxyethyl, m. 162°. <sup>224</sup>                |
| <i>i</i> -Butyl, Allyl,             | m. 147°. <sup>93, 179</sup>                            |
|                                     | <i>i</i> -Propenyl, m. 165°. <sup>54</sup>             |
|                                     | Crotyl, m. 134°. <sup>133</sup>                        |
|                                     | 2-Cyclopentenyl, m. 151°. <sup>48</sup>                |
|                                     | 2-Hydroxyethyl, m. 193°. <sup>224</sup>                |
| <i>s</i> -Butyl, Allyl,             | m. 143°. <sup>68, 235, 246</sup>                       |
|                                     | Methallyl, m. 139°. <sup>67</sup>                      |
| Amyl, Allyl,                        | m. 112.5°. <sup>179</sup>                              |
|                                     | <i>i</i> -Propenyl, m. 140°. <sup>54</sup>             |
|                                     | 2-Hydroxyethyl, m. 153°. <sup>224</sup>                |
| <i>i</i> -Amyl, <i>i</i> -Propenyl, | m. 166.5°. <sup>54</sup>                               |
|                                     | 2-Hydroxyethyl, m. 179°. <sup>224</sup>                |
|                                     | Ethylmercaptoethyl, m. 113°. <sup>263</sup>            |
| <i>s</i> -Amyl, Allyl,              | m. 133°. <sup>165</sup>                                |
|                                     | Methallyl, m. 215°. <sup>67</sup>                      |
|                                     | Methylmercaptoethyl, m. 70°. <sup>6</sup>              |
| 2-Hydroxyethyl, 2-Ethylbutyl,       | m. 165°. <sup>224</sup>                                |
|                                     | 2-Methylpentyl, m. 174°. <sup>224</sup>                |
| 2-Cyclopentenyl, 2-Bromallyl,       | m. 207°. <sup>48</sup>                                 |
|                                     | 2-Methallyl, m. 180°. <sup>48</sup>                    |
| Allyl, 1,2-Dimethylpropyl,          | m. 133°. <sup>165</sup>                                |
|                                     | 1-Ethylpropyl, m. 152°. <sup>165</sup>                 |
|                                     | Cyclohexyl, m. 150°. <sup>246</sup>                    |
|                                     | Cyclopropylmethylcarbinol, m. 139°. <sup>38</sup>      |
|                                     | Allyl, m. 134°. <sup>235, 246</sup>                    |
|                                     | Bromallyl, m. 178°. <sup>180</sup>                     |
|                                     | 2-Methallyl, m. 182°. <sup>235, 246</sup>              |
|                                     | 2-Cyclopentenyl, m. 151°. <sup>48</sup>                |

| R | R'                                       |
|---|--|
|   | 1-Cyclohexenyl, m. 170°. <sup>2</sup>    |
|   | 2-Cyclohexenyl, m. 132°. <sup>2</sup>    |
|   | 2-Cyclo-octenyl, m. 226°. <sup>143</sup> |
|   | Phenyl, m. 210°. <sup>246</sup>          |
|   | Benzyl, m. 150°. <sup>235, 246</sup>     |

## TRISUBSTITUTED-2-THIOBARBITURIC ACIDS

| R  | R'  |
|--|---|
| <i>i</i> -Propenyl, Benzyl, m. 158°. <sup>54</sup>                                 |   |
| 2-Thenyl, Ethyl, m. 217°. <sup>181</sup>   |   |
|  | <i>i</i> -Propyl, m. 199°. <sup>181</sup>     |
|  | <i>i</i> -Butyl, m. 237°. <sup>181</sup>      |
|  | <i>s</i> -Butyl, m. 195°. <sup>181</sup>      |
|  | <i>s</i> -Amyl, m. 145°. <sup>181</sup>       |
|  | 3-Methylbutyl, m. 198°. <sup>181</sup>        |
|  | Hexyl, m. 146°. <sup>181</sup>                |
|  | 1,3-Dimethylbutyl, m. 174°. <sup>181</sup>    |
|  | Heptyl, m. 130°. <sup>181</sup>               |
|  | Octyl, m. 116°. <sup>181</sup>                |
|  | 2-Ethylhexyl, m. 120°. <sup>181</sup>         |
|  | 2,2,5-Trimethylhexyl, m. 157°. <sup>181</sup> |
|  | Cyclohexyl, m. 241°. <sup>181</sup>           |
|  | Allyl, m. 179°. <sup>181</sup>                |
|  | 2-Methallyl, m. 224°. <sup>181</sup>          |
| 1,3-Diphenyl-5-aminomethylene, m. 290°. <sup>205</sup>                             |   |
| 1,3-Diphenyl-5-(2-hydroxy-1-naphthylmethylene), m. 172°. <sup>206</sup>            |   |
| 1,3-Diphenyl-5-(4-dimethylaminobenzylidene), m. above 280°. <sup>197</sup>         |   |
| 1,3-Diphenyl-5-(4-dimethylaminopiperonylidene), m. above 218°. <sup>197</sup>      |   |
| 1,3-Diphenyl-5-(4-dimethylaminovanillydene), m. 233°. <sup>197</sup>               |   |
| 1,3-Diphenyl-5-acetyl, m. 259°, <sup>217</sup> 255°. <sup>205</sup>                |   |
| 1,3-Di- <i>p</i> -tolyl-5-aminomethylene, m. 265°. <sup>205</sup>                  |   |
| 1,3-Di- <i>p</i> -tolyl-5-(2-hydroxy-1-naphthylmethylene), m. 170°. <sup>206</sup> |   |
| 5-Methyl-5-(1-cyclohexenyl)-4-imino, m. 237° dec. <sup>75</sup>                    |   |
| 5-Ethyl-5- <i>s</i> -amyl-6-imino, m. 151°. <sup>3</sup>                           |   |
| 5-Ethyl-5-(1-cyclohexenyl)-4-imino, m. 225° dec. <sup>75</sup>                     |   |
| 5- <i>s</i> -Amyl-5-(2-methylmercaptoethyl)-6-imino, m. 225°. <sup>6</sup>         |   |
| 5-Allyl-5- <i>s</i> -amyl-4-imino, m. 230°. <sup>165</sup>                         |   |
| 5-Allyl-5- <i>s</i> -amyl-6-imino, m. 231°. <sup>4</sup>                           |   |
| 5-Allyl-5-(1-ethylpropyl)-4-imino, m. 230° dec. <sup>165</sup>                     |   |

## DITHIOBARBITURIC ACIDS

- 5,5-Diethyl-2,4-dithio, m. 206°. <sup>47</sup>  
 5,5-Diethyl-4,6-dithio, m. 164°. <sup>63</sup>  
 5-Ethyl-5-propyl-2,4-dithio, m. 180°. <sup>47</sup>  
 5-Ethyl-5-*i*-propyl-4,6-dithio, m. 227°. <sup>63</sup>  
 5-Ethyl-5-butyl-2,4-dithio, m. 127°. <sup>47</sup>  
 5-Ethyl-5-*i*-butyl-2,4-dithio, m. 190°. <sup>47</sup>  
 5-Ethyl-5-*i*-butyl-4,6-dithio, m. 214°. <sup>63</sup>  
 5-Ethyl-5-*i*-amyl-4,6-dithio, m. 241°. <sup>63</sup>  
 5-Ethyl-5-benzyl-2,4-dithio, m. 180°. <sup>47</sup>  
 5,5-Dipropyl-2,4-dithio, m. 189°. <sup>47</sup>  
 5,5-Dibutyl-2,4-dithio, m. 125°. <sup>47</sup>

2,4,6-TRITHIOBARBITURIC ACIDS <sup>47</sup>

- 5,5-Diethyl, m. 193°.  
 5-Ethyl-5-propyl, m. 177°.  
 5-Ethyl-5-*i*-butyl, m. 143°.  
 5-Ethyl-5-benzyl, m. 160°.  
 5,5-Dipropyl, m. 206°.

## SPIRO THIOBARBITURIC ACIDS

- Spiro (2,2,5-Trimethylcyclopentane-2-imino-1,5'-thio), m.  
 210°. <sup>242</sup>  
 Spiro (2,2,5-Trimethylcyclopentane-1,5', 2'-thio), m. 179.5°. <sup>242</sup>  
 Spiro (2,6-Dimethylcyclohexane-1,5', 2'-thio), m. 194°. <sup>242</sup>  
 2'-Methyl-2-thiospirobarbituric acid-5,1'-cyclohexane, m.  
 172.5°. <sup>53</sup>  
 2'-Ethyl-2-thiospirobarbituric acid-5,1'-cyclohexane, m. 159.2°. <sup>53</sup>  
 2'-Propyl-2-thiospirobarbituric acid-5,1'-cyclohexane, m. 151°. <sup>53</sup>  
 6'-Methyl-2-thiospirobarbituric acid-5,1'-cyclohex-3'-ene, m.  
 143.8°. <sup>53</sup>  
 6'-Ethyl-2-thiospirobarbituric acid-5,1'-cyclohex-3'-ene, m.  
 176°. <sup>53</sup>  
 6'-Propyl-2-thiospirobarbituric acid-5,1'-cyclohex-3'-ene, m.  
 129.4°. <sup>53</sup>

## MISCELLANEOUS THIOBARBITURIC ACIDS

- 1,3-Diethyl-2-thio, m. 105°, <sup>36</sup> 103°. <sup>148</sup>  
 1,3-Diphenyl-2-thio, m. 248°. <sup>217</sup>

- 1-(3-Thenyl)-5-ethyl-2-thio, m. 171°. <sup>45</sup>  
1-(3-Thenyl)-5-allyl-2-thio, m. 155°. <sup>45</sup>  
5,5-Diethyl-S-methyl-2-thio, m. 175°. <sup>51</sup>  
5,5-Diethyl-S-carboxymethyl-2-thio, m. 170°. <sup>51</sup>  
5-Ethyl-5-(1-cyclohexenyl)-S-methyl-2-thio, m. 175°. <sup>51</sup>  
5-Ethyl-5-(1-cyclohexenyl)-S-carboxymethyl-2-thio, m. 155°. <sup>51</sup>

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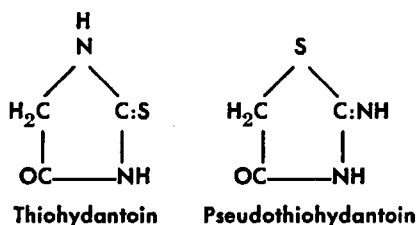
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## CHAPTER 5

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# Thiohydantoin and Pseudothiohydantoin

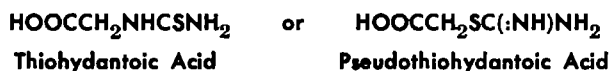
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### Introduction

These are considered together as they both contain the same ring elements and both can be obtained from the same starting materials under only slightly different reaction conditions. Pseudothiohydantoin has the thiazole ring structure but is isomeric with thiohydantoin with which it has been closely associated and often confused. The two formulae are alike except that the sulfur atom and the imino group have exchanged places. In pseudothiohydantoin the sulfur atom is a member of the ring, while in thiohydantoin it is not and it can be removed without breaking the ring. In reading the older literature one has to look carefully into the reactions to be sure which is meant. Much painstaking work was required to differentiate the two compounds and to determine their structures.<sup>5, 62, 84, 85, 163, 175, 193, 196, 197, 201, 298, 344, 351, 352</sup>

According to whether the chloracetic acid reacts with an amino group or with the sulfur atom of the thiourea, the product would be:



These condense readily with the elimination of water to form the cyclic structures given at the beginning. The rates of condensation of a number of substituted thiohydantoic acids have been measured.<sup>70</sup> In fact the condensation takes place so readily that these acids are seldom isolated. When a thiohydantoic acid is desired it is usually prepared by the hydrolysis of the corresponding thiohydantoin.<sup>48, 120</sup> Both of these acids are thiourea derivatives and are prepared accordingly. If we wish to obtain a given thiohydantoin we employ the reactants and the methods suitable for the synthesis of the thiohydantoic acid, which usually condenses under the conditions of its formation. To obtain substituted thiohydantoins it is convenient to start with the properly substituted reactants. Similar statements can be made about the synthesis of pseudothiohydantoins.

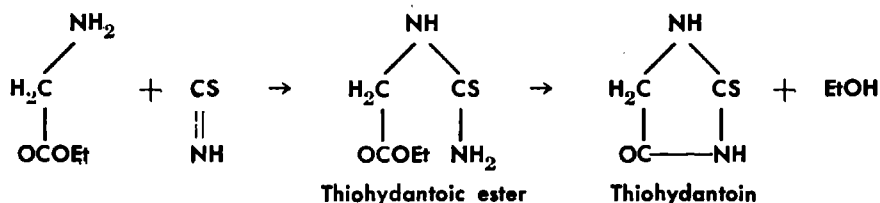
Another reason for considering the thiohydantoins and the pseudothiohydantoins together is that many of their reactions are practically the same. This is particularly true of those that concern the 5-position, the hydrogens of which are readily replaced; condensations with aldehydes and ketones take place readily.

### Thiohydantoin

Thiohydantoin forms yellow prisms, m. 228°, with decomposition. It is slightly soluble in hot alcohol and in hot water.

### PREPARATION

The first preparation was from ethyl aminoacetate hydrochloride and potassium thiocyanate: <sup>131, 156, 175</sup>



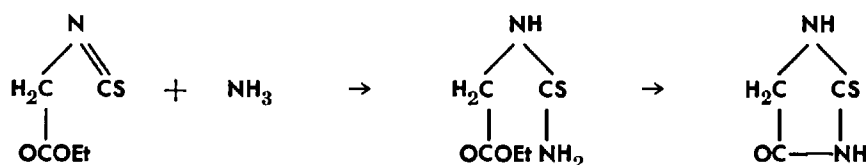
In this preparation an ester was used, but the amino acid or its amide serves as well. Thiohydantoic acid is formed when aminoacetic acid, or its ester, is heated with potassium thiocyanate in acetanhydride. It is converted into thiohydantoin by hydrochloric acid.<sup>180</sup> Acetylglycine,  $\text{MeCONHCH}_2\text{CO}_2\text{H}$ , has been considered to be an intermediate<sup>180</sup> but this has been doubted. It does yield 1-acetyl-2-thiohydantoin with potassium thiocyanate and this is deacetylated by hydrochloric acid.<sup>156, 165, 168</sup> Acetylthiohydantoic acid,  $\text{MeCONHCSNHCH}_2\text{CO}_2\text{H}$ , or its ester, is converted into thiohydantoin by digestion with hydrochloric acid.<sup>305</sup> The ethyl ester of N-cyanoethylglycine and phenyl isothiocyanate condense to the ester of a thiohydantoic acid.<sup>276</sup> Butyryl ethyldithiocarbamate combines with glycine, but the resulting thiohydantoic acid cannot be cyclized because of the butyryl group.<sup>9</sup> Hippuric acid,  $\text{PhCONHCH}_2\text{CO}_2\text{H}$ , which is benzoylglycine, and potassium or ammonium thiocyanate give 1-benzoyl-2-thiohydantoin<sup>158, 267, 340</sup> which can be debenzoylated to 2-thiohydantoin.<sup>158</sup> The ethyl ester of N-aminoglycine and potassium thiocyanate give 1-amino-2-thiohydantoin.<sup>289</sup> 1-Phenyl-2-thiohydantoin is formed from N-phenyl glycine and ammonium thiocyanate.<sup>98</sup> Ammonium thiocyanate gives better yields than potassium or sodium thiocyanate.<sup>156, 165</sup> It may be that the amino acid replaces the ammonia. The thiocyanate of ethyl aminoacetate is transformed by acid catalysis to 2-thiohydantoin.<sup>105</sup>

A number of N-acylamino acids have been converted into the corresponding 1-acyl-2-thiohydantoins which may be hydrolyzed to 2-thiohydantoins.<sup>168, 182, 209, 267, 273, 274, 300, 308</sup> Radioactive N-acylamino acids have been prepared by the use of radioactive ammonium thiocyanate.<sup>282</sup>

Glycine,  $\text{CH}_2(\text{NH}_2)\text{COOH}$ , the simplest  $\alpha$ -amino acid, gives thiohydantoin; thiohydantoins having substituents in the 5-position are derived from other  $\alpha$ -amino acids,  $\text{RCH}(\text{NH}_2)\text{COOH}$ . Many of these have been prepared.<sup>6, 7, 46, 49, 103, 116, 121, 146, 149, 161, 164, 170, 180, 211, 245, 304</sup>

The  $\alpha$ -anilide of acetyl-DL-aspartic acid and ammonium thiocyanate give the 1-acetyl-2-thio-5-hydantoin acetanilide; the propionanilide is obtained from the corresponding acetylamino glutaric acid.<sup>267</sup> Reduced glutathione gives a bithiohydantoin.<sup>209</sup>

The addition of ammonia to an ester of isothiocyanoacetic acid gives a thiourea which condenses to 2-thiohydantoin: <sup>162</sup>



Substitution of ethyl aminoacetate for the ammonia in the preceding leads to 2-thio-hydantoin-3-acetic ester.<sup>167</sup>

N-Alkylthiocarbamyl glycines are cyclized to 3-substituted-2-thiohydantoins by acid.<sup>151</sup> N-Phenyl-N-benzoylthiocarbamyl glycine is cyclized by concentrated sulfuric acid to 1-phenyl-3-benzoylthiohydantoin, whereas with dilute sulfuric acid the benzoyl group is lost.<sup>104</sup>

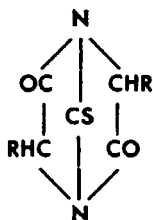
The addition of an alkyl or aryl isothiocyanate to an  $\alpha$ -amino acid gives a 3-substituted 2-thiohydantoin.<sup>144</sup> An N-substituted dithiocarbamate may supply the necessary isothiocyanate.<sup>177</sup> Methyl isothiocyanate and glycine unite to form the thiourea,  $\text{MeNHCSNHCH}_2\text{CO}_2\text{H}$ , which condenses to 3-methyl-2-thiohydantoin. With alanine the product is the 3,5-dimethyl- and with  $\alpha$ -aminobutyric acid it is 3,5,5-trimethylthiohydantoin. Allyl mustard oil reacts similarly.<sup>199</sup> Methyl isothiocyanate and hydrazinoacetic acid give 1-amino-3-methyl-2-thiohydantoin.<sup>280</sup> Phenyl isothiocyanate has been combined with several  $\alpha$ -amino acids to make 3-phenyl-5-R-2-thiohydantoins.<sup>35, 96, 113, 148, 251</sup> Amino acids are conveniently identified by means of their addition products with phenyl<sup>106, 190, 251</sup> or substituted phenyl<sup>106, 227</sup> isothiocyanates.

In the stepwise degradation of a peptide it is treated with phenyl isothiocyanate and the cleavage products are converted to thiohydantoins.<sup>77, 95, 306, 322</sup> The 3-substituted thiohydantoins can be separated by chromatography<sup>98, 251</sup> or paper chromatography<sup>184</sup> and identified by their infrared spectra;<sup>106, 224</sup> or the thiohydantoin content may be determined by ultraviolet absorption measurements.<sup>322</sup> Methyl, butyl, and benzyl isothiocyanates have been used for the same purpose as the phenyl compound.<sup>104</sup> Similar syntheses have been made with other amino acids and isothiocyanates.<sup>12, 13, 14, 15, 16, 78, 84, 93, 130, 139, 169, 177, 183, 208, 225, 233, 279, 305</sup> Benzoyl isothiocyanate is converted into the corresponding 3-benzoyl-2-thiohydantoins.<sup>102</sup> Cysteic acid and methyl acetyldithiocarbamate, passed through Dowex 50 resin

and hydrolyzed with acid, gives 5-sulfomethyl-2-thiohydantoin.<sup>103</sup>

Diethylglycolic acid and thiourea condense to 5,5-diethyl-2-thiohydantoin.<sup>60</sup> Benzil and thiourea give 5,5-diphenyl-2-thiohydantoin, one of the phenyl groups migrates as in the pinacolone rearrangement.<sup>29, 123, 124</sup> Unsymmetrical compounds,  $\text{RCOCOPh}$ , react similarly; a substituent in the thiourea goes into the 3-position.<sup>118</sup>  $\alpha$ -Pyridil can be converted to 5,5-di(2-pyridyl)-2-thiohydantoin by reaction with thiourea; substituted thioureas give the corresponding 3-substituted.<sup>176, 277</sup>

Phenyl thiochloracetate,  $\text{ClCH}_2\text{COSPh}$ , forms 2-thiohydantoin with the elimination of thiophenol.<sup>71</sup> 1,3-Disubstituted-2-thiohydantoins are formed from symmetrical disubstituted thioureas and chloracetic acid.<sup>221</sup> They are also obtained from N-substituted glycines and alkyl or aryl isothiocyanates.<sup>108, 143, 153</sup> Carbon disulfide and  $\alpha$ -amino-acids react to form thioureas,  $\text{SC}(\text{NHCHRCO}_2\text{H})_2$  which can be condensed to double thiohydantoins:<sup>179</sup>



The ester-thiourea,  $\text{H}_2\text{NCSNHCOOEt}$ , is condensed by alkali to 2-thiohydantoin.<sup>160</sup>

Several 5-aminothiazoles have been isomerized to 2-thiohydantoins.<sup>47, 63</sup> A 2-thiohydantoin can be obtained from a thiazoline<sup>64, 65</sup> or a thiazolidone.<sup>69, 142</sup>

A ketone, sodium cyanide, and ammonium thiocarbamate give a 5,5-disubstituted-4-thiohydantoin.<sup>51</sup> Cyanomethyl ammonium cyanomethylthiolcarbamate in acetone is converted by hydrochloric acid to 4-thio-5-isopropylidenehydantoin.<sup>66</sup>

### DITHIOHYDANTOINS

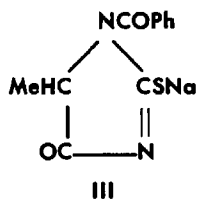
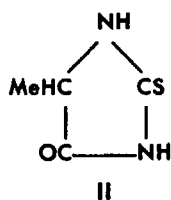
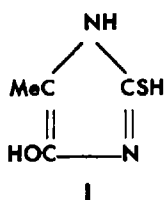
Dithiohydantoins may be prepared by sulfurizing 2-thiohydantoins<sup>36</sup> or hydantoins<sup>138, 260</sup> with phosphorus trisulfide or pentasulfide, or by the reaction of carbon disulfide with an  $\alpha$ -amino-

nitrile.<sup>53, 65, 150</sup> 1,5,5-Trimethylhydantoin, refluxed with sulfur and phosphorus pentasulfide in xylene, gives 1,5,5-trimethyl-2,4-dithiohydantoin.<sup>49</sup>  $\alpha$ -Cyanocyclohexylamine and carbon disulfide give pentamethylene-5,5-spiro-2,4-dithiohydantoin.<sup>50</sup> Cyclooctanone, sodium cyanide, ammonium chloride, carbon disulfide, ethanol, and water, heated in an autoclave, yield 2,4-dithio-5,5-heptamethylene spirohydantoin.<sup>228</sup>

The first product may be a 5-amino-2-mercaptothiazole which rearranges to the dithiohydantoin.<sup>67, 68, 75, 212, 312</sup> An  $\alpha$ -aminoamide,  $\text{H}_2\text{NCH}_2\text{CONH}_2$ , may take the place of the aminonitrile.<sup>67</sup> A 5-substituted 2,4-dithiohydantoin may be prepared by treating a cyanhydrin with carbon disulfide in the presence of ammonia.<sup>238</sup> A ketone-cyanhydrin,  $\text{Me}_2\text{C}(\text{OH})\text{CN}$ , gives 5,5-dimethyl-2,4-dithiohydantoin.<sup>135</sup> The ketone, ammonium chloride, sodium cyanide, and carbon disulfide may be heated together in aqueous methanol.<sup>49, 107</sup> The melting points of some 5,5-derivatives, prepared in this way, are given in the lists of physical properties. A cyanhydrin may be combined with hydrogen sulfide and the resulting thioamide treated with  $\text{HCNS}$  to give the dithiohydantoin.<sup>107</sup> Triethylammonium [1-cyano-3-(methylthio)propyl] dithiocarbamate, heated in water solution is converted to 5-[2-(methylthio)ethyl]-2,4-dithiohydantoin.<sup>142</sup> Refluxing a solution of potassium cyanide, ammonium chloride, acetone, carbon disulfide, and ethanol, then adding alkali and acidifying, gives 5,5-dimethyl-2,4-dithiohydantoin which is desulfurized to the 4-monothio by silver oxide.<sup>147</sup>

### KETO-ENOL ISOMERISM

By condensing unsymmetrical dihydroxyacetone with thiourea, 2-thio-4-hydroxy-5-methylimidazoline, I, was prepared and found to be identical with the well known 5-methyl-2-thiohydantoin, II.



The II prepared from *l*-alanine was optically active but was racemized when it was dissolved in sodium hydroxide, indicating a shift to the enol form I. The 1-benzoyl derivative from benzoyl-*l*-alanine was optically active and remained so in the presence of one equivalent of alkali but was racemized. This can be explained by assuming that the mono-sodium salt has the structure III.<sup>252</sup> Alkylation of I is considered under reactions.

### SPECTROSCOPIC PROPERTIES

The substitution of sulfur for the oxygen of a hydantoin causes a general shift of the absorption to longer wave length.<sup>265</sup> The ultraviolet absorption spectra and paper chromatographic behavior of various substituted 2-thiohydantoins were studied for the purpose of identification.<sup>102</sup> The spectra were determined in alcohol, water, and aqueous sodium hydroxide; 1- and 3-substitution can be distinguished.<sup>98</sup> The difference in the spectrum of benzoylthiohydantoin in alcohol from that in water is attributed to hydrolysis.<sup>281</sup>

Infrared spectra of a number of thiohydantoins have been investigated.<sup>101, 224</sup> In some cases the locations of the CO bands of thiohydantoins and of their benzylidene derivatives are different and in others they are not.<sup>313</sup>

### REACTIONS

The polarographic behavior of dimethyldithiohydantoin has been studied.<sup>239, 240, 241</sup>

A thiohydantoin is hydrolyzed by barium or potassium hydroxide to a thiohydantoic acid.<sup>15, 60, 175, 248</sup> The thiohydantoic acid can be desulfurized by mercuric oxide<sup>15, 175</sup> and recondensed to a hydantoin by boiling with hydrochloric acid.<sup>15</sup> More vigorous hydrolysis of the thiohydantoin yields thiourea.<sup>248</sup> A study has been made of the rates of hydrolysis of 3-phenyl-5-R-2-thiohydantoins.<sup>189</sup>

A thiohydantoin is readily desulfurized to the corresponding hydantoin by bromine,<sup>14, 15</sup> nitric acid, or alkaline permanganate,<sup>29</sup> also by boiling with chloracetic<sup>158, 166, 339</sup> or hydrochloric acid.<sup>49</sup>

Hydrogenolysis transforms substituted thiohydantoins into de-



rivatives of 4-oxotetrahydroglyoxaline.<sup>51</sup> Refluxing 5,5-diphenyl-2-thiohydantoin with sodium in *i*-amyl alcohol removes the sulfur and puts hydrogen in its place, forming 5,5-diphenyl-4-oxotetrahydroglyoxaline.<sup>30</sup> Raney nickel gives a variety of desulfurization products.<sup>19, 97, 259, 260, 303</sup> 2-Thio- and 2,4-dithiohydantoins are desulfurized by copper or its salts.<sup>237</sup>

5,5-Dimethyl-2,4-dithiohydantoin is completely desulfurized by refluxing with dilute hydrochloric acid. The sulfur in the 4-position is removed by treatment with a primary amine. Hydrolysis of the imino derivative gives the 2-thiohydantoin.<sup>49</sup> Treatment with aniline or with a hydrazine replaces the sulfur by a substituted imino group.<sup>299</sup>

Deacylation of 1-acyl 2-thiohydantoins takes place rapidly, being practically complete in five minutes in 0.1N alkali.<sup>174</sup> In an attempted deacylation by aqueous hydrazine the products turned out to be  $\alpha$ -thioureidohydrazides.<sup>98</sup>

A thiohydantoin is alkylated on the sulfur by an alkyl halide in the presence of alkali.<sup>32, 165, 180</sup> When 5,5-diphenyl-2-thiohydantoin is methylated strongly the second methyl group goes onto the nitrogen in the 1-position.<sup>54</sup> 5-Methyl-2-thiohydantoin would be expected to shift to the enol form in the presence of excess alkali, but when it is methylated the second methyl goes to the 5-position, giving the 5,5-dimethyl- which is identical with the authentic 5,5-dimethyl compound.<sup>265</sup> This is analogous to the alkylation of acetoacetic ester. The alkyl in the 2-position is removed as a mercaptan by acid hydrolysis, leaving a hydantoin. An alkyl in the 1-position is not affected. Thus a 1,2-dialkylthiohydantoin yields a 1-alkylhydantoin.<sup>54</sup>

Methylation of 2,4-dithiohydantoin puts methyl groups in the 1- and 3-positions,<sup>75</sup> but with its 5,5-dimethyl derivative methylation takes place on the sulfur.<sup>137</sup> Refluxing a 2,4-dithiohydantoin with ethanolamine replaces the 4-sulfur by the imino group; hydrolysis substitutes the keto group for the imino.<sup>52</sup> The addition of iodine to 2,5,5-trimethyldithiohydantoin and of methyl iodide to 5,5-dimethyl-dithiohydantoin give the same product.<sup>55</sup>

### CONDENSATION WITH ALDEHYDES

Thiohydantoins that have no substituents in the 5-position condense with aldehydes.<sup>20, 34, 48, 82, 83, 93, 134, 155, 159, 168, 207, 209, 289, 290, 292, 304</sup> The condensation has been effected in acetan-

hydride containing sodium acetate, in which case there may also be acetylation.<sup>82, 207</sup> Condensation also takes place in acetic acid with sodium acetate<sup>93</sup> or in pyridine solution with a secondary amine as catalyst.<sup>34</sup> An aldehyde derivative such as the phenyl-hydrazone may be substituted for the free aldehyde.<sup>209</sup> The aldehyde derivatives are well characterized compounds which may be alkylated<sup>304</sup> and desulfurized<sup>82, 168</sup> by the same methods as their parent thiohydantoins.

#### ANALYTICAL

Alkyl thiohydantoin may be determined colorimetrically<sup>236</sup> and various 5-substituted thiohydantoins fluorimetrically.<sup>11</sup> Phosphotungstic and phosphotungstic-phosphomolybdic acid solutions give color reactions.<sup>326, 345</sup>

Some derivatives of 2-thiohydantoin are sensitive reagents for heavy metals. The 5,5-diphenyl- is recommended for cobalt ions. It can be used as a precipitant in the gravimetric quantitative analysis of copper, nickel, and cobalt ions.<sup>302</sup> The cobalt salt has received particular attention.<sup>89, 90, 91, 119</sup> Several benzylidene derivatives have been recommended as reagents for a number of heavy metals.<sup>284</sup> Dimethyl dithiohydantoin has been proposed as a reagent for copper.<sup>45</sup> Phenyl<sup>61, 69.5, 124.5, 221</sup> and substituted phenyl<sup>61</sup> thiohydantoic acids have been suggested for the detection of cobalt and other metals.

#### PHYSIOLOGICAL

2-Thiohydantoin is toxic to rabbits; the 4-methyl derivative is less so.<sup>191</sup> 2-Thiohydantoin,<sup>57, 154, 172, 200</sup> its 5-benzal,<sup>8</sup> 5-(2-propylidene),<sup>133</sup> and other<sup>172, 187, 249</sup> derivatives have been tested for their antithyroid activity. A bis-thiohydantoin<sup>58</sup> and several derivatives of 2,4-dithiohydantoin<sup>57, 218, 229, 293</sup> have been found to be active. Thiohydantoin has been compared with thiouracil<sup>261</sup> and it and its benzoyl and acetyl derivatives with thiourea<sup>185</sup> as antithyroids. Dimethyldithiohydantoin has been found to have remarkable antiepileptic properties<sup>136</sup> but may cause myxedema.<sup>87</sup> 5,5-Diphenyl-2-thiohydantoin has also been used in an antiepileptic preparation.<sup>141</sup> 5-Ethyl-5-phenyl-2-thiohydantoin combats epilepsy but is too toxic for medical use.<sup>31</sup> The 5,5-dimethyl-<sup>59, 135, 223</sup> and 5-methyl-5-ethyl-2,4-dithiohy-

dantoin<sup>135</sup> give efficient protection against convulsions. The sodium salt of 5,5-diphenylthiohydantoin and several dithiohydantoins have been tested as anticonvulsants.<sup>124, 137</sup> 5-(2-Cyclohexylethyl)-2-thiohydantoin is said to be useful for its anti-tubercular properties.<sup>6</sup> In rabbits dimethyldithiohydantoin and methylenedithiohydantoin were antagonistic to curare-like drugs, but increased the effects of acetylcholine neuromuscular blocking agents.<sup>56</sup> 2-Thiohydantoin-5-acetic acid inhibits the growth and blocks lactic acid production in cultures of *Lactobacillus casei*.<sup>255</sup> Pretreatment with 5-isopropylidene-2,4-dithiohydantoin increased the resistance of rats to intravenously administered alloxan. It also decreased the incidence of diabetes caused by the removal of 95% of the pancreas.<sup>145</sup> 5-Isopropylidene-2,4-dithiohydantoin inhibited insulin degradation in rats.<sup>100</sup> Several thiohydantoins have been found to possess antibacterial properties.<sup>291</sup> 2-Thiohydantoin has been fed to dogs and its degradation products determined in the urine.<sup>140</sup>

#### APPLICATIONS

Some thiohydantoins have been used in copper plating baths.<sup>43, 132</sup> Of much greater importance is the use of various substituted thiohydantoins in photography.<sup>1, 2, 10, 38, 39, 41, 80, 81, 88, 111, 117, 125, 126, 127, 152, 178, 194, 230, 250, 258, 262, 295, 296</sup>

### Pseudothiohydantoin

#### PREPARATION

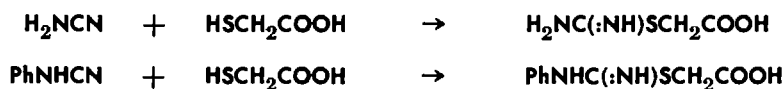
The relationship of pseudothiohydantoin to thiohydantoin has been discussed in the general introduction. As stated there, pseudothiohydantoin is obtained by the cyclization of pseudothiohydanitoic acid,  $\text{HOOCCH}_2\text{SC}(\text{:NH})\text{NH}_2$ , a 1-carboxymethyl thiourea. In general the synthesis of a substituted pseudothiohydantoin is effected by bringing together the reactants necessary to produce the corresponding pseudothiohydanitoic acid, which is seldom isolated as it is cyclized under the same conditions under which it is formed.

Pseudothiohydantoin is monoclinic, space group  $\text{P2}_1/\text{a}$ , 4 mols. per unit cell whose dimensions are  $a = 13.79$ ,  $b = 9.00$ ,  $c = 4.04$  Å.,  $\beta = 110.0^\circ$ . The crystals show negative birefringence with  $n_\alpha = 1.54$ ,  $n_\beta = 1.74$ ,  $n_\gamma = 1.91$ , optic angle  $2V = 70^\circ$ .  $\beta$  is par-

allel to  $b$ , and  $\gamma$  is approximately parallel to  $a$ . The observed density is 1.637; the calculated density, 1.635.<sup>217</sup> Absorption maxima are given for 5-benzylidene pseudothiohydantoin.<sup>265</sup> Its heat of combustion at constant volume is 502.4 K cal./mol.<sup>201</sup> It forms salts with auric and platinic chlorides and with lead oxide.<sup>246</sup>

A 2-keto-4-imino compound, m.  $71^\circ$ , has been prepared and named isothiohydantoin.<sup>333</sup>

Another method of making pseudothiohydantoin, which throws light on its structure, is the addition of thioglycolic acid to cyanamide<sup>5</sup> or one of its substitution products:<sup>188</sup>



By the loss of water these go into pseudothiohydantoin and its 3-phenyl derivative. Active and racemic thiolactic acids give the same 5-methylpseudothiohydantoin.<sup>188</sup>

Pseudothiohydantoin is readily prepared, in high yields, by heating thiourea with chloracetic acid,<sup>198, 203, 297</sup> its sodium salt,<sup>196</sup> an ester, or an amide.<sup>256, 257</sup> The first reaction is the formation of the isothiuronium salt, the hydrochloride of pseudothiohydantoic acid, which is condensed to the pseudothiohydantoin by further heating. Either the salt or the acid may be isolated.<sup>256, 257</sup> One method is to reflux for three hours a solution of 76 g. thiourea and 125 g. ethyl chloracetate in 500 cc. of alcohol. The crude hydrochloride so formed is dissolved in 1200 cc. of water, and 121 g. crystallized sodium acetate, dissolved in 150 cc. of water, is added. The yield is about 80%.<sup>4</sup> Pseudothiohydantoin is also formed by heating methyl isothiurea with mercaptoacetic acid in benzene.<sup>112</sup>

The use of a mono-substituted thiourea leads to a pseudothiohydantoin with a substituent in the 2-position.<sup>73, 76, 94, 109, 110, 218, 231, 233, 234, 272, 307, 349</sup> The two groups of a symmetrical thiourea take the 2- and 3-positions.<sup>3, 5, 21, 22, 25, 26, 85, 128, 193, 204, 226, 243, 294</sup> The acetyl group of  $\text{Ph}_2\text{NCSNHAc}$  and the benzoyl of  $\text{Ph}_2\text{NC-SNHCOPh}$  are eliminated in the condensation.<sup>86</sup>

Thiourea and  $\alpha$ -halogen acids give pseudothiohydantoins with substituents in the 5-position.<sup>5, 62, 72, 79, 109, 110, 210, 220, 235, 244, 253, 266, 301</sup> Ethyl  $\alpha, \alpha'$ -dibromosuberate and thiourea give 5,5'-tetramethylene bis-pseudothiohydantoin.<sup>215</sup> The thiocyano-acid,

RCH(SCN)COOH, condenses to the pseudothiohydantoin.<sup>328, 349</sup>

A double compound is obtained from  $\text{H}_2\text{NCSNH}\cdot\text{NHCSNH}_2$ .<sup>115</sup>

The ureide of an  $\alpha$ -halogen acid serves as well. Thus  $\alpha$ -bromo-*i*-valeric ureide gives *i*-propylpseudothiohydantoin urea, the  $-\text{CONH}_2$  group being in the 3-position.<sup>176.5</sup>

Thiourea acts like a mercaptan in adding to maleic, fumaric, and  $\beta$ -benzoyl-acrylic acids. The addition products condense to pseudothiohydantoins substituted in the 5-position.<sup>5, 33, 157, 315</sup> Citroconic acid gives pseudothiohydantoin-5- $\alpha$ -propionic acid with thiourea, and the dimethyl derivative of this acid with *s*-dimethylthiourea.<sup>5</sup>

1,2-Dichlorovinyl ethyl ether and phenyl thiourea in acetone give 2-phenyl pseudothiohydantoin.<sup>271</sup> A methanol solution of thiourea and ethyl 1-oxaspiro[2,5]-octane-2-carboxylate form in three weeks 5-(1-hydroxycyclohexyl)pseudothiohydantoin.<sup>109</sup> *p*- $\text{ClCH}_2\text{CONHC}_6\text{H}_4\text{SO}_2\text{NH}_2$  and potassium thiocyanate yield 3(*p*-sulfamylphenyl)pseudothiohydantoin.<sup>270</sup>

2-Imino-3,5-diphenyl-4-amino-4-thiazoline hydrochloride is hydrolyzed to 3,5-diphenyl-pseudothiohydantoin.<sup>275</sup> The sodium salt of 3-phenyl-2-ethoxy-2-mercapto-oxazolidone-4 reacts with chloracetanilide to give the 2,3-diphenylpseudothiohydantoin.<sup>336</sup>

## REACTIONS

The rates of alkaline hydrolysis of several substituted pseudothiohydantoins have been compared.<sup>287</sup> Solution in alkali causes decomposition.<sup>342</sup> The complete hydrolysis of a pseudothiohydantoin gives an  $\alpha$ -mercapto-acid corresponding to the  $\alpha$ -halogen-acid that was used in its synthesis. Thus thioglycolic acid is obtained from the unsubstituted pseudothiohydantoin.<sup>5, 62, 84, 193, 210, 244, 301</sup> Certain groups in the 5-position stabilize pseudothiohydantoins against bisulfite.<sup>285</sup>

Oxidation breaks the ring.<sup>321</sup> Acetylurea sulfonic acid,  $\text{H}_2\text{NCONHCOCH}_2\text{SO}_3\text{H}$ , is the first product and sulfoacetic acid,  $\text{HOOCCH}_2\text{SO}_3\text{H}$ , the next.<sup>5, 173</sup> Urea oxalic acid and sulfuric acid may be produced.<sup>246</sup>

As with the thiohydantoins, condensation with an aldehyde takes place if there is no substituent in the 5-position.<sup>23, 24, 25, 26, 28, 73, 74, 109, 128, 181, 192, 222, 226, 233, 234, 242, 288, 297, 309, 314</sup> The condensation may take place along with the formation of the pseudo-

thiohydantoin. Thus, chloracetic acid, thiourea, and benzaldehyde, heated in acetic acid, give the 5-benzylidene derivative.<sup>297</sup> A derivative of this class has been prepared by condensing the aldehyde with mustard oil acetic acid and converting this product to the pseudothiohydantoin.<sup>181</sup> Condensation with ketones has also been effected.<sup>109, 269</sup>

Alkylation of the silver salt has given confusing results. It is claimed that alkylation in sodium hydroxide solution, in the presence of copper powder, introduces an alkyl into the 5-position. This suggests keto-enol isomerization as with the thiohydantoins.<sup>5, 254, 311</sup> Alkylation of pseudothiohydantoin-aldehyde condensation products can be effected by alcoholic potash and alkyl iodide.<sup>73</sup>

As was stated in the preceding section, thiohydantoin is readily desulfurized. This is far from being the case with pseudothiohydantoin. It gives up little sulfur when boiled with water and mercuric oxide;<sup>206</sup> after four weeks at 100° the removal is not complete. However, the sulfur can be taken out by freshly precipitated mercuric oxide in ammonium hydroxide. The ring is broken up by hydrolysis and oxidation with the formation of guanidine and oxalic acid.<sup>246</sup> It will be recalled that the removal of the sulfur of thiohydantoin does not disturb the ring.

5-Isonitrosopseudothiohydantoin is obtained by treatment with nitrous acid.<sup>5, 196, 197</sup> This is a very weak acid yet its sodium salt is neutral.<sup>129</sup> It forms characteristic salts with heavy metals and has been recommended as an analytical reagent.<sup>91, 92</sup> With barium hydroxide it gives the barium salt of nitrosothioglycolic acid.<sup>5</sup>

Pseudothiohydantoins can be mercurated by mercuric acetate in acetic acid.<sup>73, 195, 222</sup>

A 5-arylazo derivative can be obtained by coupling a pseudothiohydantoin with a diazonium chloride.<sup>27, 218, 219, 233</sup>

Aniline in boiling water replaces the 2-imino group of a pseudothiohydantoin by the phenylimino.<sup>109, 198</sup> Heating auramine with 3-phenyl pseudothiohydantoin forms a dye.<sup>214</sup> 5-Benzylidene-2-(*p*-chlorophenylimino)thiazolidinone dibromide is obtained from 5-benzylidene-2-(*p*-chlorophenylimino)-4-thiazolidinone, chloroform, and bromine.<sup>222</sup> Quinaldine-MeI and 5-hydroxyimino-pseudothiohydantoin produce a black crystalline compound.<sup>314</sup>

## ANALYTICAL

Analytical procedures have been given for the detection and identification of pseudothiohydantoin.<sup>286</sup> 5-(*p*-Dimethylamino-benzylidene)-3,2'-diphenylpseudothiohydantoin is a reagent for silver and thallium; 5-benzylidene-3-allylpseudothiohydantoin is a reagent for bismuth.<sup>283</sup>

## PHYSIOLOGICAL

Pseudothiohydantoin has antithyroid action corresponding closely to that of thiouracil.<sup>232</sup> It causes an increase in the weight of the thyroid of rats.<sup>17</sup> Several substituted pseudothiohydantoins have been tested for antitubercular activity.<sup>99, 202, 205</sup> Some have antibacterial activity.<sup>195, 222, 272.5</sup> Mercurated derivatives have been tested as bactericides<sup>195, 222</sup> and fungicides.<sup>195, 222</sup> Pseudothiohydantoin is a hypertensive agent.<sup>247</sup> It does not affect the basal metabolism of white mice.<sup>122</sup> Two of its derivatives inhibit the evolution of carbon dioxide from a yeast-glucose fermentation.<sup>114</sup> Its 5,5-dimethyl derivative has no anticonvulsive effect.<sup>135</sup>

## APPLICATIONS

Pseudothiohydantoin has been used as the basis in the synthesis of photographic dyes.<sup>37, 151</sup> Its copper salt is a good gelatin activator for photographic emulsions.<sup>263</sup> Several of its derivatives are ultraviolet absorbers,<sup>242, 243</sup> and one is said to protect plastics against weathering.<sup>268</sup> One pseudothiohydantoin is recommended as an activator with a thiazole accelerator.<sup>18</sup>

**Physical Properties of 2-Thiohydantoin and Some of Its  
Substitution Products**

2-Thiohydantoin, m. 228° dec.,<sup>71</sup> 227°,<sup>19, 165, 267, 305</sup> 227–9° dec.,<sup>105</sup>  
222–4° dec.,<sup>104</sup> picrate, m. 195–8°.<sup>71</sup>

## 1-SUBSTITUTED 2-THIOHYDANTOIN

Acetyl, m. 179°,<sup>182, 267</sup> 176°.<sup>19, 156, 165</sup>

Benzoyl, m. 165° dec.<sup>156</sup>

*m*-Nitrobenzoyl, m. 199°.<sup>168</sup>

Anisoyl, m. 166°.<sup>168</sup>

N-Benzoylglycyl, m. 210–15°. <sup>341</sup>  
 N-Benzoyl-DL-alanyl, m. 188°. <sup>341</sup>  
 N-Benzoyl-DL-leucyl, m. 172°. <sup>341</sup>  
 Phenylsulfonyl, m. 180°, <sup>304</sup> 175°. <sup>104</sup>  
*p*-Tolylsulfonyl, m. 235°. <sup>287</sup>  
 Carbobenzyloxy, m. 186°. <sup>287</sup>  
 Amino, m. 188° dec., <sup>290</sup> 191°; <sup>48</sup> HCl, m. 168° dec. <sup>290</sup>  
 2-Furfurylidine amino, m. 218°. <sup>291</sup>  
 5-Nitro-2-furfurylidine amino, m. 232°. <sup>289, 291</sup>  
 2-Furylvinyl CH:N-, m. 238° dec. <sup>291</sup>  
 5-Nitro-2-furylvinyl CH:N-, m. 257° dec. <sup>291</sup>  
 5-Nitro-2-thenylidene amino, m. 236–9°. <sup>48</sup>  
 Benzylidene amino, m. 233°, <sup>290</sup> 227–9°. <sup>48</sup>  
*p*-NO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>CH:N, m. 262°. <sup>292</sup>  
*p*-AcNHC<sub>6</sub>H<sub>4</sub>CH:N, m. 272°. <sup>292</sup>  
*p*-MeOC<sub>6</sub>H<sub>4</sub>CH:N, m. 243°. <sup>292</sup>  
*p*-EtSO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>CH:N, m. 270°. <sup>292</sup>  
*p*-HOCC<sub>6</sub>H<sub>4</sub>CH:N, m. 285°. <sup>292</sup>  
 Methyl, m. 224–6°. <sup>103</sup>  
 Phenyl, m. 180°, <sup>304</sup> 175°. <sup>104</sup>

### 3-SUBSTITUTED-2-THIOHYDANTOIN

Benzoyl, m. 155–7°. <sup>104</sup>  
 Methyl, m. 170° and 162°, <sup>151</sup> 161°. <sup>199</sup>  
 Ethyl, m. 203°; <sup>151</sup> deuteriated ethyl, m. 141°. <sup>101</sup>  
 Octyl, m. 192°. <sup>151</sup>  
*c*-Hexyl, m. 231°. <sup>151</sup>  
 Allyl, m. 108°. <sup>199</sup>  
 Phenyl, dec. about 200°. <sup>317</sup>  
 Benzyl, m. 196°, <sup>151</sup> 128°. <sup>166</sup>  
 Tolyl, *o*, m. 150°, <sup>166</sup> 136°; <sup>199</sup> *p*, m. 228°, <sup>166</sup> 210°, <sup>199</sup> 170°. <sup>151</sup>  
*o*-Nitrophenyl, m. 164°. <sup>225</sup>  
*p*-Phenylazophenyl, m. 180°. <sup>225</sup>  
 HOCC<sub>6</sub>H<sub>4</sub>, m. 138°. <sup>151</sup>

### 5-SUBSTITUTED-2 THIOHYDANTOIN

Methyl, m. 163–5°, <sup>149</sup> 161°, <sup>144</sup> 157°. <sup>104</sup>  
 Ethyl, m. 164–6°. <sup>149</sup>  
 Propyl, m. 156.5°. <sup>149</sup>  
*i*-Propyl, m. 160°, <sup>267</sup> 138–40°, <sup>149</sup> 237–9°. <sup>312</sup>



- Butyl, m.  $135.4^{\circ}$ ,<sup>149</sup>  $131^{\circ}$ .<sup>104</sup>  
*i*-Butyl, m.  $174-6^{\circ}$ ,<sup>149</sup>  $173^{\circ}$ .<sup>267</sup>  
*s*-Butyl, m.  $131-3^{\circ}$ .<sup>149</sup>  
 Amyl, m.  $132^{\circ}$ .<sup>149</sup>  
 Hexyl, m.  $144.5^{\circ}$ .<sup>149</sup>  
 Heptyl, m.  $131.6^{\circ}$ .<sup>116</sup>  
*c*-Hexylmethyl, m.  $225.7^{\circ}$ .<sup>149</sup>  
*c*-Hexylethyl, m.  $177.8^{\circ}$ .<sup>6</sup>  
 2-Thenyl, m.  $198^{\circ}$  dec.<sup>46</sup>  
 3-Thienyl, m.  $178^{\circ}$ .<sup>46</sup>  
 Phenyl, m.  $250^{\circ}$  dec.,<sup>274</sup>  $227^{\circ}$ ,<sup>303</sup>  $223-5^{\circ}$ .<sup>267</sup>  
 Benzyl, m.  $184.5^{\circ}$ ,<sup>149</sup>  $176^{\circ}$ .<sup>104</sup>  
*p*-Hydroxybenzyl, m.  $206^{\circ}$ .<sup>144</sup>  
 MeSCH<sub>2</sub>, m.  $170^{\circ}$ .<sup>103</sup>  
 MeSCH<sub>2</sub>CH<sub>2</sub>, m.  $151^{\circ}$ ,<sup>149</sup>  $149^{\circ}$ .<sup>267</sup>  
 EtSCH<sub>2</sub>CH<sub>2</sub>, m.  $100.5^{\circ}$ .<sup>46</sup>  
 MeSO<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>, m.  $228^{\circ}$ .<sup>103</sup>  
 Benzoylaminomethyl, m.  $255^{\circ}$  dec.<sup>209</sup>  
 3-Aminopropyl, m.  $210^{\circ}$ .<sup>103</sup>  
 3-Guanidinopropyl, HCl, m.  $209^{\circ}$ .<sup>103</sup>  
 4-Aminobutyl, m.  $235-7^{\circ}$ ,<sup>103</sup> Ac., m.  $194^{\circ}$ .<sup>267</sup>  
 BuCOCH<sub>2</sub>, m.  $88.7-101.3^{\circ}$ .<sup>6</sup>  
 AmCOCH<sub>2</sub>, m.  $98.2-101.2^{\circ}$ .<sup>6, 7</sup>  
 HexCOCH<sub>2</sub>, m.  $101.5^{\circ}$ ,<sup>7</sup>  $100^{\circ}$ .<sup>6</sup>  
 HeptCOCH<sub>2</sub>, m.  $111.8^{\circ}$ ,<sup>7</sup>  $104-108.9^{\circ}$ .<sup>6</sup>  
 OctCOCH<sub>2</sub>, m.  $110.7-12.7^{\circ}$ .<sup>6, 7</sup>  
 NonCOCH<sub>2</sub>, m.  $110.3-130^{\circ}$ .<sup>6</sup>  
 PrCOCH<sub>2</sub>CH<sub>2</sub>, m.  $121.9^{\circ}$ .<sup>6</sup>  
 BuCOCH<sub>2</sub>CH<sub>2</sub>, m.  $115.6^{\circ}$ .<sup>6, 7</sup>  
 AmCOCH<sub>2</sub>CH<sub>2</sub>, m.  $107.7^{\circ}$ .<sup>6, 7</sup>  
 EtOOC, m.  $227^{\circ}$ .<sup>267</sup>  
 H<sub>2</sub>NCOCH<sub>2</sub>, m.  $246^{\circ}$ .<sup>267</sup>  
 H<sub>2</sub>NCOCH<sub>2</sub>CH<sub>2</sub>, m.  $191^{\circ}$ ,<sup>267</sup>  $189^{\circ}$ ,<sup>103</sup> anilide, m.  $216^{\circ}$ ,<sup>209</sup>  $215^{\circ}$ .<sup>267</sup>  
 3-Indolyl, m.  $191^{\circ}$ .<sup>267</sup>  
 4-Glyoxalinylmethyl, HCl, m.  $285-8^{\circ}$ .<sup>103</sup>

### 1,3-DISUBSTITUTED 2-THIOHYDANTOIN

- 1            3  
 Acetyl, *p*-Tolyl, m.  $157^{\circ}$ .<sup>151</sup>  
           NH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>-, m.  $135-7^{\circ}$ .<sup>132</sup>

| 1   | 3 |
|---|---|
| Amino, Methyl, m. 120°. <sup>280</sup>  |   |
| Phenyl, m. 165°. <sup>280</sup>   |   |
| 5-Nitrofurfurylidene amino, Methyl, m. 218° dec. <sup>134</sup>                                     |   |
| Ureido, Phenyl, m. 211°. <sup>319</sup>   |   |
| 3,3'-Ethylene-bis-(1-methyl-2-thiohydantoin), m. 201°. <sup>151</sup>                               |   |
| Methyl, Methyl, m. 141°. <sup>177</sup> 94.5°. <sup>78</sup>  |   |
| Ethyl, m. 69.5°. <sup>101</sup> 67°. <sup>151</sup>   |   |
| Heptyl, b <sub>2</sub> 190°. <sup>151</sup>   |   |
| c-Hexyl, m. 156°. <sup>151</sup>  |   |
| Phenyl, m. 163°. <sup>65</sup>  |   |
| p-Tolyl, m. 149°. <sup>151</sup>  |   |
| 2-Thiazolyl, m. 133°. <sup>177</sup>  |   |
| Ethyl, Methyl, m. 151-3°. <sup>12</sup>   |   |
| Ethyl, m. 85°. <sup>177</sup>   |   |
| p-HOCC <sub>6</sub> H <sub>4</sub> , m. 219.5°. <sup>153</sup>                                      |   |
| 2-Thiazolyl, m. 177°. <sup>177</sup>  |   |
| 2-Pyridyl, m. 132°. <sup>177</sup>  |   |
| Propyl, Ethyl, m. 144-6°. <sup>12</sup>   |   |
| Heptyl, Heptyl, m. 70°. <sup>177</sup>  |   |
| c-Hexyl, c-Hexyl, m. 174°. <sup>177</sup>   |   |
| Allyl, Allyl, m. 62°. <sup>177</sup>  |   |
| Phenyl, Methyl, m. 113.5°. <sup>93</sup>  |   |
| Ethyl, m. 125.5-28.5°. <sup>42</sup> 100°. <sup>12</sup>  |   |
| Phenyl, m. 235°. <sup>124</sup> 212°. <sup>316, 318</sup> 186°. <sup>177</sup> 174°. <sup>221</sup> |   |
| 2-Pyridyl, m. 190°. <sup>177</sup>  |   |
| Benzyl, Methyl, m. 74°. <sup>12</sup>   |   |
| Ethyl, m. 82°. <sup>12</sup>  |   |
| Phenyl, m. 189.5°. <sup>279</sup>   |   |
| p-Tolyl, p-Tolyl, m. 115°. <sup>221</sup>   |   |
| α-Naphthyl, α-Naphthyl, m. 183°. <sup>221</sup>   |   |
| 3,3'-Ethylene-bis-1-Methyl-2-thiohydantoin, m. 201°. <sup>151</sup>                                 |   |
| 2-Pyridyl, Methyl, m. 141°. <sup>177</sup>  |   |
| Ethyl, m. 107°. <sup>177</sup>  |   |
| Heptyl, m. 70°. <sup>177</sup>  |   |
| c-Hexyl, m. 174°. <sup>177</sup>  |   |
| Allyl, m. 62°. <sup>177</sup>   |   |
| Phenyl, m. 186°. <sup>177</sup>   |   |
| Benzyl, m. 115°. <sup>177</sup>   |   |
| 2-Pyridyl, m. 173°. <sup>177</sup>  |   |

| 1   | 3  |
|---|--|
|   | 6-Methyl-2-pyridyl, m. 147°. <sup>177</sup>    |
|   | 4-Methyl-2-pyridyl, m. 151°. <sup>177</sup>    |
|   | 3-Pyridyl, m. 171°. <sup>177</sup>             |
|   | 3-Quinolyl, m. 204°. <sup>177</sup>            |
| 6-Methyl-2-pyridyl, Ethyl, m. 85°. <sup>177</sup>   |  |
|   | 6-Methyl-2-pyridyl, m. 219–21°. <sup>177</sup> |
| 3-Pyridyl, 3-Pyridyl, m. 171°. <sup>177</sup>   |  |
| 4-Methyl-3-pyridyl, 4-Methyl-3-pyridyl, m. 151°. <sup>177</sup>                           |  |
| 3-Quinolyl, 3-Quinolyl, m. 204°. <sup>177</sup>   |  |
| Carboxymethyl, Ethyl, m. 160–3°; Et ester, m. 86°. <sup>108</sup>                         |  |
| Caboxyphenyl, Phenyl, m. 278–80°; <sup>108</sup> Et ester, m. 188–90°. <sup>40, 108</sup> |  |

## 1,5-DISUBSTITUTED-2-THIOHYDANTOIN

| 1  | 5 |
|--|---|
| Acetyl, Methyl, m. 166°, <sup>182</sup> 164°. <sup>144</sup>                                       |   |
| <i>i</i> -Propyl, m. 113°; <sup>182</sup> DL, m. 114°. <sup>174</sup>                              |   |
| Butyl, m. 163°. <sup>182</sup>   |   |
| <i>i</i> -Butyl, m. 129°. <sup>182</sup>   |   |
| <i>s</i> -Butyl, DL, m. 153.8°. <sup>174</sup>   |   |
| Phenyl, m. 193°. <sup>274</sup>  |   |
| Benzyl, m. 170°. <sup>182</sup>  |   |
| <i>p</i> -Hydroxybenzyl, m. 144°. <sup>273</sup>   |   |
| <i>p</i> -Acetoxybenzyl, m. 192–4°. <sup>273</sup>   |   |
| 4,3,5-(HO)Br <sub>2</sub> C <sub>6</sub> H <sub>2</sub> CH <sub>2</sub> , m. 197°. <sup>273</sup>  |   |
| 4,3,5-(AcO)Br <sub>2</sub> C <sub>6</sub> H <sub>2</sub> CH <sub>2</sub> , m. 186°. <sup>273</sup> |   |
| 4,3,5-(AcO)I <sub>2</sub> C <sub>6</sub> H <sub>2</sub> CH <sub>2</sub> , m. 167°. <sup>273</sup>  |   |
| 2-Thenyl, m. 168°. <sup>46</sup>   |   |
| 3-Thienyl, m. 158°. <sup>46</sup>  |   |
| HSCH <sub>2</sub> -, m. 208°. <sup>182</sup>   |   |
| AcSCH <sub>2</sub> -, m. 142°. <sup>182</sup>  |   |
| MeSCH <sub>2</sub> CH <sub>2</sub> -, m. 104°; <sup>182</sup> DL, m. 106°. <sup>174</sup>          |   |
| MeSO <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> -, m. 195.5°. <sup>103</sup>                     |   |
| MeOCHMe-, m. 152–60°. <sup>308</sup>   |   |
| <i>p</i> -HOC <sub>6</sub> H <sub>4</sub> CH <sub>2</sub> -, m. 248°. <sup>182</sup>               |   |
| 3-Indolylmethyl, m. 170°. <sup>182</sup>   |   |
| EtOOC-, m. 109°. <sup>267</sup>  |   |
| H <sub>2</sub> NCOCH <sub>2</sub> -, m. 224°, <sup>182</sup> 219°. <sup>267</sup>                  |   |
| PhNHCOCH <sub>2</sub> -, m. 242°. <sup>267</sup>   |   |
| MeOCOCH <sub>2</sub> CH <sub>2</sub> -, m. 102°. <sup>103</sup>                                    |   |

- 1                      5  
 $\text{H}_2\text{NCOCH}_2\text{CH}_2-$ , m.  $219^\circ$ ,<sup>182</sup>  $209^\circ$ .<sup>209</sup>  
 $\text{PhNHCOCH}_2\text{CH}_2-$ , m.  $197^\circ$ .<sup>209, 267</sup>  
 Phenylacetyl,  $\text{EtOOC}-$ , m.  $110^\circ$ .<sup>267</sup>  
 $\text{H}_2\text{NCOCH}_2\text{CH}_2-$ , m.  $177^\circ$ ; <sup>267</sup> D, m.  $162^\circ$ ;  $[\alpha]^{20}$   $118.5^\circ$ ;  
 inactive, m.  $169^\circ$ .<sup>211</sup>  
 Benzoyl, Benzoylaminomethyl, m.  $212^\circ$  dec.<sup>209</sup>  
*p*-Nitrobenzoyl,  $\text{PhNHCOCH}_2\text{CH}_2-$ , m.  $200^\circ$ .<sup>267</sup>  
 N-benzoylglycyl, *i*-Propyl, m.  $186-9^\circ$ .<sup>341</sup>  
*i*-Butyl, m.  $186^\circ$ .<sup>341</sup>  
 $\text{PhCH}_2\text{OOC}-$ , Methyl, m.  $192^\circ$ .<sup>300</sup>  
 $\alpha$ -Methyl-5-nitro-2-furfurylidene amino-, Methyl, m.  $225^\circ$  dec.<sup>134</sup>  
 5,5'-Dithiodimethylene-*bis*-1-acetyl-2-thiohydantoin, m.  $198-202^\circ$ .<sup>267</sup>

## 3,5-DISUBSTITUTED-2-THIOHYDANTOIN

- 3                      5  
 Acetyl,  $\text{EtSCH}_2\text{CH}_2-$ , m.  $58.5^\circ$ .<sup>46</sup>  
*p*- $\text{ClC}_6\text{H}_4-$ , m.  $168^\circ$ .<sup>46</sup>  
*p*- $\text{BrC}_6\text{H}_4-$ , m.  $188.5^\circ$ .<sup>46</sup>  
 Methyl, Methyl, m.  $168^\circ$ .<sup>144</sup>  
*i*-Butyl, m.  $150^\circ$ .<sup>144</sup>  
 $\text{MeSCH}_2\text{CH}_2-$ , m.  $81^\circ$ .<sup>144</sup>  
*p*- $\text{HOC}_6\text{H}_4\text{CH}_2-$ , m.  $260^\circ$ .<sup>144</sup>  
 $\text{HOOCCH}_2\text{CH}_2-$ , m.  $146^\circ$ .<sup>144</sup>  
 Ethyl, 4,3,5-(HO) $\text{Br}_2\text{C}_6\text{H}_2\text{CH}_2$ , m.  $190-2^\circ$ .<sup>273</sup>  
 Allyl, 4,3,5-(HO) $\text{Br}_2\text{C}_6\text{H}_2\text{CH}_2$ , m.  $200-2^\circ$ .<sup>273</sup>  
 Phenyl, *i*-Butyl, m.  $175^\circ$ .<sup>144</sup>  
 $\text{HOCH}_2-$ , m.  $176-8^\circ$ .<sup>148</sup>  
 $\text{HOCH}_2\text{CH}_2-$ , m.  $194^\circ$  dec.<sup>148</sup>  
 $\text{H}_2\text{NCOCH}_2\text{CH}_2-$ , m.  $201-11^\circ$ .<sup>96</sup>  
 $\text{HOOCCH}_2\text{SCH}_2-$ , m.  $164^\circ$ .<sup>96</sup>  
 Potassium methyl sulfonate, m.  $193-7^\circ$ .<sup>96</sup>  
 4,3,5-(HO) $\text{I}_2\text{C}_6\text{H}_2\text{CH}_2$ , m.  $164-6^\circ$ .<sup>273</sup>  
*o*-Nitrophenyl, *i*-Propyl, m.  $159-62^\circ$ .<sup>225</sup>  
 Benzyl, m.  $140^\circ$ .<sup>225</sup>  
 $\text{HSCH}_2-$ , m.  $113^\circ$ .<sup>225</sup>  
*p*- $\text{HOC}_6\text{H}_4\text{CH}_2-$ , m.  $191^\circ$ .<sup>225</sup>  
 $\text{HOOCCH}_2-$ , m.  $210^\circ$ ; amide, m.  $236^\circ$ .<sup>225</sup>  
 $\text{HOOCCH}_2\text{CH}_2-$ , m.  $166-8^\circ$ .<sup>225</sup>

- 3                      5  
*p*-PhN:NC<sub>6</sub>H<sub>4</sub>, Methyl, m. 184–6°. <sup>225</sup>  
                          *i*-Propyl, m. 259°. <sup>225</sup>  
                          *i*-Butyl, m. 221°. <sup>225</sup>  
                          Benzyl, m. 255°. <sup>225</sup>  
                          MeSCH<sub>2</sub>CH<sub>2</sub>-, m. 211°. <sup>225</sup>  
                          HOCH<sub>2</sub>-, m. 152°. <sup>225</sup>  
                          HOCHMe-, m. 234°. <sup>225</sup>  
                          *p*-HOC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>-, m. 225°. <sup>225</sup>  
                          H<sub>2</sub>NC(:NH)NHCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>-, m. 182°. <sup>225</sup>  
                          HOOCCH<sub>2</sub>-, m. 224°. <sup>225</sup>  
                          HOOCCH<sub>2</sub>CH<sub>2</sub>-, m. 197°; amide, m. 211°. <sup>225</sup>  
                          HOOCCH<sub>2</sub>-, *i*-Propyl, m. 168°. <sup>121</sup>

#### 5,5-DISUBSTITUTED-2-THIOHYDANTOIN

- Methyl, Methyl, m. 179°, <sup>49</sup> 174°. <sup>107, 135</sup>  
                          *i*-Butyl, m. 152°. <sup>51</sup>  
                          Phenyl, m. 189°. <sup>51</sup>  
                          Acetylamino, m. 248° dec. <sup>209</sup>  
                          Ethyl, Phenyl, m. 170–2°, <sup>303</sup> 171°. <sup>51</sup>  
                          *c*-Hexyl, *c*-Hexyl, m. 189°. <sup>135</sup>  
                          Phenyl, Phenyl, m. 240°, <sup>44</sup> 234°. <sup>123</sup>  
                          5,6,7,8-Tetrahydro-2-naphthyl, m. 191°. <sup>118</sup>  
                          2-Pyridyl, 2-Pyridyl, m. 258–60°, <sup>176</sup> 255°. <sup>277</sup>  
                          5,5-*c*-Tetramethylene, m. 198°. <sup>51</sup>  
                          5,5-*c*-Pentamethylene, m. 192°. <sup>49</sup>  
                          5,5-*c*-(3-Methyl-pentamethylene), m. 219°. <sup>49</sup>  
                          5,5-*c*-(2-Methylhexamethylene), m. 157°. <sup>36</sup>  
                          5,5-*c*-(3-Methylhexamethylene), m. 147–50°. <sup>36</sup>  
                          5,5-*c*-Heptamethylene, m. 204°. <sup>337</sup>  
                          5,5-*c*-(*o*-CH<sub>2</sub>C<sub>6</sub>H<sub>10</sub>CH<sub>2</sub>CH<sub>2</sub>-), m. 206°. <sup>36</sup>

#### TRISUBSTITUTED 2-THIOHYDANTOINS

- 1,3,5-Triphenyl, m. 162°. <sup>143</sup>  
                          1,5-Trimethylene-3-methyl, m. 52°. <sup>101</sup>  
                          1-Acetyl-5-methyl-5-acetylamino, m. 230°. <sup>209</sup>  
                          1-Acetyl-5,5-dimethyl, m. 187°. <sup>49</sup>  
                          1-Acetyl-5,5-pentamethylene, m. 165–7°. <sup>49</sup>  
                          1,5-Dimethyl-5-phenyl, m. 197°. <sup>51</sup>

- 3-Acetyl-5-methyl-5-phenyl, m.  $174^{\circ}$ .<sup>46</sup>  
 3-Methyl, 5-Phenyl, 5-methyl, m.  $224^{\circ}$ .<sup>51</sup>  
     5-phenyl, m.  $215^{\circ}$ .<sup>52</sup>  
     5-(5,6,7,8-tetrahydro-  
       2-naphthyl), m.  $210^{\circ}$ .<sup>118</sup>  
     5,5-Di(2-pyridyl), m.  $194-6^{\circ}$ ,<sup>176</sup>  $194^{\circ}$ .<sup>277</sup>  
 3-c-Hexyl, 5,5-Di(2-pyridyl), m.  $218^{\circ}$ .<sup>277</sup>  
 3-Allyl, 5,5-Di(2-pyridyl), m.  $197^{\circ}$ .<sup>277</sup>  
 3-Phenyl, 5,5-Di(2-pyridyl), m.  $240^{\circ}$  dec.<sup>277</sup>  
 3-Benzyl, 5,5-Di(2-pyridyl), m.  $200^{\circ}$ .<sup>176</sup>  
     5,5-Diphenyl, m.  $153.5^{\circ}$ .<sup>183</sup>

## TETRASUBSTITUTED 2-THIOHYDANTOINS

- 1,3,5-Trimethyl-5-phenyl, m.  $112^{\circ}$ .<sup>51</sup>  
 1,3-Dimethyl-5,5-diphenyl, m.  $144^{\circ}$ .<sup>52</sup>

## 4-SUBSTITUTED-2-THIOHYDANTOIN

- 4-(2-Furfurylidene), m.  $250^{\circ}$ .<sup>69</sup>  
 5,5-Dimethyl, 4-imino, m. about  $353^{\circ}$  dec.,<sup>107, 135</sup>  $300-2^{\circ}$ .<sup>42</sup>  
     4-( $\text{:NCH}_2\text{CH}_2\text{OH}$ ), m.  $224^{\circ}$ .<sup>49</sup>  
     4-isonitroso, m.  $250^{\circ}$ ,<sup>135</sup>  $242^{\circ}$ .<sup>107</sup>  
     4-hydrazono, m.  $220^{\circ}$ ,<sup>107, 135</sup>  $188^{\circ}$  dec.<sup>49</sup>  
     4- $\text{:N:N:CH}_2$ , m.  $217^{\circ}$ .<sup>107</sup>  
     4- $\text{:N:N:CMe}_2$ , m.  $233^{\circ}$ .<sup>107</sup>  
     4- $\text{:N:N:CMeEt}$ , m.  $196^{\circ}$ .<sup>107</sup>  
     4- $\text{:N:N:CMeCH}_2\text{COOEt}$ , m.  $95^{\circ}$ .<sup>209</sup>  
 5-Methyl, 5-Phenyl, 4- $\text{:NCH}_2\text{CH}_2\text{OH}$ , m.  $201^{\circ}$ .<sup>51</sup>  
 5-Ethyl, 5-Phenyl, 4- $\text{:NCH}_2\text{CH}_2\text{OH}$ , m.  $185^{\circ}$ .<sup>51</sup>  
 5,5-Diphenyl, 4- $\text{:NCH}_2\text{CH}_2\text{OH}$ , m.  $218^{\circ}$  (0.5 mol.  $\text{H}_2\text{O}$ ).<sup>52</sup>  
 5,5-Di-c-hexyl, 4-imino, m.  $293^{\circ}$ .<sup>135</sup>  
 5,5-c-Pentamethylene, 4- $\text{:NCH}_2\text{CH}_2\text{OH}$ , m.  $232^{\circ}$ ,<sup>51</sup>  $245^{\circ}$ .<sup>49</sup>  
 5,5-c-3-Methylpentamethylene, 4-( $\text{:NCH}_2\text{CH}_2\text{OH}$ ), m.  $234^{\circ}$ .<sup>49</sup>  
      $\text{Me}_2\text{CNH}\cdot\text{CS}\cdot\text{NHC:N}(\text{CH}_2)_n\text{N:C}\cdot\text{NH}\cdot\text{CS}\cdot\text{NH}\cdot\text{CMe}_2$ .  
      $n=0$ , m.  $241^{\circ}$ .<sup>107</sup>  $n=5$ , m.  $308^{\circ}$ .<sup>58</sup>  
      $n=2$ , m.  $235^{\circ}$ .<sup>58</sup>  $n=6$ , m.  $294^{\circ}$ .<sup>58</sup>  
      $n=3$ , m.  $255^{\circ}$ .<sup>58</sup>  $n=10$ , m.  $277^{\circ}$ .<sup>58</sup>  
 1-Methyl-5,5-c-pentamethylene-4-( $\text{:NCH}_2\text{CH}_2\text{OH}$ ), m.  $159^{\circ}$  (2  
   mols.  $\text{H}_2\text{O}$ ).<sup>52</sup>  
 1,3-Dimethyl-5,5-diphenyl-4-( $\text{:NCH}_2\text{CH}_2\text{OH}$ ), m.  $58^{\circ}$ .<sup>52</sup>

## 2-SUBSTITUTED-2-THIOHYDANTOIN

- 5,5-Dimethyl, 2-methiodide, m.  $204^{\circ}$ .<sup>135</sup>  
     2-methyl, m.  $195^{\circ}$ ; 4-imino compd., m.  $217^{\circ}$ .<sup>135</sup>  
 5,5-Diphenyl, 2-methyl, m.  $205^{\circ}$ .<sup>54</sup>  
     2-ethyl, m.  $180^{\circ}$ .<sup>54</sup>  
     2-benzyl, m.  $190^{\circ}$ .<sup>54</sup>  
     2-thioglycolic acid, m.  $195^{\circ}$ .<sup>32</sup>  
 5,5-Di-*p*-methoxyphenyl, 2-benzyl, m.  $148^{\circ}$ .<sup>54</sup>  
 1-Methyl-5,5-diphenyl, 2-methyl, m.  $172^{\circ}$ .<sup>54</sup>  
 1-Ethyl-5,5-diphenyl, 2-ethyl, m.  $97^{\circ}$ .<sup>54</sup>  
 1-Benzyl-5,5-diphenyl, 2-benzyl, m.  $84^{\circ}$ .<sup>54</sup>  
 1-Benzyl-5,5-di-*p*-methoxyphenyl, 2-benzyl, m.  $131^{\circ}$ .<sup>54</sup>  
 $(\text{NH}\cdot\text{CPh}_2\cdot\text{CO}\cdot\text{N}\cdot\text{CS})_2\text{Co}$ , m.  $226-8^{\circ}$ ;  $2\text{NH}_3$  compd., m.  $228^{\circ}$ .<sup>119</sup>

## ALDEHYDE DERIVATIVES OF 2-THIOHYDANTOIN

*Aldehyde (or ketone)*

- Acetaldehyde, m.  $264^{\circ}$ .<sup>267</sup>  
 Acetone, m.  $258-60^{\circ}$ .<sup>312</sup>  
*i*-Butyraldehyde, m.  $194^{\circ}$ ,<sup>19</sup>  $174-6^{\circ}$ .<sup>312</sup>  
 MePrCO, m.  $152^{\circ}$ .<sup>312</sup>  
 MeBuCO, m.  $112-14^{\circ}$ .<sup>312</sup>  
 MeAmCO, m.  $115^{\circ}$ .<sup>312</sup>  
 MeHeptylCO, m.  $183-5^{\circ}$ .<sup>312</sup>  
*c*-Pentanone, m.  $252^{\circ}$ .<sup>69</sup>  
*c*-Hexanone, m.  $263-5^{\circ}$ .<sup>312</sup>  
 3-Me-*c*-hexanone, m.  $240-2^{\circ}$ .<sup>312</sup>  
 4-Me-*c*-hexanone, m.  $245-7^{\circ}$ .<sup>312</sup>  
 Benzal, m.  $259^{\circ}$ ,<sup>207</sup>  $258^{\circ}$ ,<sup>267</sup>  $257^{\circ}$ ; <sup>312</sup> Ac., m.  $260^{\circ}$ .<sup>207</sup>  
 Hydroxybenzaldehyde, *o*, m.  $231^{\circ}$ ; diAc., m.  $237^{\circ}$ ; *m*, m.  $250^{\circ}$ ;  
     diAc., m.  $250^{\circ}$ ; <sup>207</sup> *p*, m.  $305^{\circ}$ ,<sup>34</sup>  $270^{\circ}$ ; diAc., m.  $265^{\circ}$ .<sup>207</sup>  
 3,5-Dihydroxybenzaldehyde, m.  $210^{\circ}$ ; triAc., m.  $240^{\circ}$ .<sup>207</sup>  
*p*-Methoxybenzaldehyde, m.  $255^{\circ}$ ; Ac., m.  $265^{\circ}$ .<sup>207</sup>  
 3,4-Methylenedioxybenzaldehyde, m.  $288-90^{\circ}$ ,<sup>82</sup>  $283^{\circ}$ ; Ac., m.  
      $275^{\circ}$ .<sup>207</sup>  
 Vanillal, m.  $240^{\circ}$ ; diAc., m.  $261^{\circ}$ .<sup>207</sup>  
 Nitrobenzaldehyde, *o*, m.  $249^{\circ}$ ; Ac., m.  $241^{\circ}$ ; *m*, m.  $257^{\circ}$ ; Ac., m.  
      $263^{\circ}$ ; *p*, m.  $266^{\circ}$ ; Ac., m.  $270^{\circ}$ .<sup>207</sup>  
*p*-Chlorobenzaldehyde, m.  $274^{\circ}$  dec.<sup>19</sup>

- p*-Me<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO, m. 261°, <sup>69</sup> 252°; Ac., m. 272°. <sup>207</sup>  
 Cinnamaldehyde, m. 260°; Ac., m. 267°. <sup>207</sup>  
 $\beta$ -Indolylidene, m. 312–20° dec. <sup>19</sup>  
 4-Glyoxalinylmethyl, HCl, m. 285–8° dec. <sup>103</sup>  
 Pyrrolidino[1':2',1:5], m. 161–3°. <sup>103</sup>

#### ALDEHYDE OR KETONE DERIVATIVES OF SUBSTITUTED 2-THIOHYDANTOIN

Substituents: *Aldehyde or Ketone*

- 3-Methyl, PhCHO, m. 207°, <sup>69</sup> 204°, <sup>63</sup> 201°. <sup>93</sup>  
     *o*-BrC<sub>6</sub>H<sub>5</sub>CHO, m. 170°. <sup>155</sup>  
 3-(EtO)<sub>2</sub>CHCH<sub>2</sub>, Acetone, m. 88°. <sup>171</sup>  
 3-Phenyl, Me<sub>2</sub>CO, m. 254–7° dec. <sup>64</sup>  
     Me<sub>2</sub>CHCHO, m. 209°. <sup>69</sup>  
     PhCHO, *trans.*, m. 207°; *cis*, m. 160°. <sup>69</sup>  
     *p*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO, m. 266°. <sup>69</sup>  
 1,3-Dimethyl, PhCHO, m. 139°. <sup>265</sup>  
 1,3-Diethyl, (3-Et-4,5-diPh-2-oxazolinylidene), m. 182°. <sup>151</sup>  
     (3-Bu-4,5-bis-*p*-MeOC<sub>6</sub>H<sub>4</sub>-2-oxazolinylidene-ethyl-  
     idene), m. 186°. <sup>151</sup>  
 1-Phenyl, 3-Methyl, PhCHO, m. 138°. <sup>93</sup>  
     3-Ethyl, *p*-Me<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO, m. 204°. <sup>42</sup>  
     (1-Et-4-pyridylidene), m. 243–5°. <sup>186</sup>

#### Physical Properties of 4-Thiohydantoin and Some of Its Substitution Products

##### 4-THIOHYDANTOIN

- 4-Thiohydantoin, dec. 220°; <sup>160</sup> 5-Me<sub>2</sub>C: deriv., m. 227–9°; <sup>66</sup>  
     N-phenylisonitroso deriv., m. 210–12°. <sup>173</sup>  
 5,5-Dimethyl, m. 240°. <sup>135</sup>  
 5,5-Methyl, ethyl, m. 233°. <sup>51</sup>  
 5,5-Methyl, *i*-Butyl, m. 199°. <sup>51</sup>  
 5,5-Methyl, hexyl, m. 156°. <sup>51</sup>  
 5,5-Methyl, phenyl, m. 238°. <sup>51</sup>  
 5,5-Methyl, benzyl, m. 217°. <sup>51</sup>  
 5,5-Diphenyl, m. 274°. <sup>52</sup>  
 5,5-(CH<sub>2</sub>)<sub>4</sub>-, m. 206°. <sup>51</sup>  
 5,5-(CH<sub>2</sub>)<sub>5</sub>-, m. 243°, <sup>51</sup> 240°. <sup>49</sup>  
 5,5-CHMe(CH<sub>2</sub>)<sub>4</sub>-, m. 252°. <sup>51</sup>



- 5,5-CH<sub>2</sub>CHMe(CH<sub>2</sub>)<sub>3</sub>-, m. 279°. <sup>51</sup>  
 5,5-(CH<sub>2</sub>)<sub>2</sub>CHMe(CH<sub>2</sub>)<sub>2</sub>-, m. 262°. <sup>51</sup>  
 5,5-CH<sub>2</sub>CH(CHMe<sub>2</sub>)(CH<sub>2</sub>)<sub>3</sub>-, m. 262°. <sup>51</sup>  
 5,5-CH<sub>2</sub>CHMeCH<sub>2</sub>CMe<sub>2</sub>CH<sub>2</sub>-, m. 225°. <sup>51</sup>  
 5,5-CH<sub>2</sub>CHMeCH<sub>2</sub>CHMeCH<sub>2</sub>-, m. 330°. <sup>51</sup>  
 5,5-(CH<sub>2</sub>)<sub>2</sub>CHMeCHMeCH<sub>2</sub>-, m. 275°. <sup>51</sup>  
 5,5-(CH<sub>2</sub>)<sub>6</sub>-, m. 178°. <sup>51</sup>  
 5,5-CHMe(CH<sub>2</sub>)<sub>5</sub>-, m. 249°. <sup>51</sup>  
 3,5-Dimethyl-5-phenyl, m. 130°. <sup>51</sup>  
 5,5-Diphenyl-3-methyl, m. 202°. <sup>52</sup>  
 1-Methyl-5,5-pentamethylene, m. 189°. <sup>52</sup>  
 1,3-Dimethyl-5,5-diphenyl, m. 211°. <sup>52</sup>  
 1,3-Dimethyl-5,5-pentamethylene, m. 119°. <sup>52</sup>  
 $\overline{\text{HN}\cdot\text{C}(\text{:NCH}_2\text{CH}_2\text{OH})\text{NH}\cdot\text{CS}\cdot\text{CR}_2}$ , diphenyl, m. 239°; penta-  
 methylene, m. 194°. <sup>52</sup>  
 S<sup>4</sup>,5,5-Trimethyl, m. 233°. <sup>135</sup>

### Physical Properties of 2,4-Dithiohydantoin and Some of Its Substitution Products

#### 2,4-DITHIOHYDANTOIN

- 2,4-Dithiohydantoin, 1,3-diAc., m. 161°; <sup>67</sup> 5-*p*-Me<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CH:  
 5-*p*-Me<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CH:, m. 269° dec.; <sup>314</sup> 5-Me<sub>2</sub>C:, m. 235°. <sup>67</sup>

#### 5-SUBSTITUTED DITHIOHYDANTOIN

- Methyl-, Ac., m. 165°. <sup>67</sup>  
*i*-Propyl-, m. 232°. <sup>75</sup>  
 Hexyl-, Ac., m. 155°. <sup>68</sup>  
 1-Ethylamyl-, m. 258°. <sup>68</sup>  
 Octyl-, 1,3-diAc., m. 189°. <sup>312</sup>  
 Phenyl-, m. 265°. <sup>67</sup>  
 Propenyl-, m. 231-4°. <sup>212</sup>  
 MeSCH<sub>2</sub>CH<sub>2</sub>-, m. 222°, <sup>142</sup> 212° dec. <sup>53</sup>  
 EtSCH<sub>2</sub>CH<sub>2</sub>-, m. 197° dec. <sup>53</sup>  
 EtOOC(CH<sub>2</sub>)<sub>4</sub>-, m. 200°. <sup>68</sup>

#### DISUBSTITUTED DITHIOHYDANTOIN

- 1,3-diMethyl-, m. 90°. <sup>75</sup>  
 1 (or 3)-Benzyl, 5-Phenyl-, m. 194°. <sup>67</sup>

## 5,5-DISUBSTITUTED DITHIOHYDANTOIN

Methyl, Methyl, m.  $148^{\circ}$ .<sup>138</sup>  $144^{\circ}$ .<sup>49</sup>  $143^{\circ}$ .<sup>147, 150</sup>  $142^{\circ}$ .<sup>135</sup> 2-methiodide, m.  $133^{\circ}$ .<sup>135</sup> 4-methiodide, m.  $183^{\circ}$ .<sup>107</sup> 4,4-diiodo, m.  $135^{\circ}$ .<sup>55</sup>

Ethyl, m.  $148^{\circ}$ .<sup>49</sup>

Propyl, m.  $124^{\circ}$ .<sup>49</sup>

*i*-Propyl, m.  $226^{\circ}$ .<sup>49</sup>

*i*-Butyl, m.  $92^{\circ}$ .<sup>49</sup>  $90^{\circ}$ .<sup>51</sup>

*t*-Butyl, m.  $236^{\circ}$ .<sup>49</sup>

Amyl, m.  $118^{\circ}$ .<sup>49</sup>

*i*-Hexenyl, m.  $99^{\circ}$ .<sup>49</sup>

*c*-Hexyl, m.  $196^{\circ}$ .<sup>49</sup>

Phenyl, m.  $177^{\circ}$ .<sup>138</sup>

Benzyl, m.  $156^{\circ}$ .<sup>49</sup>

Phenethyl, m.  $180^{\circ}$ .<sup>49</sup>

Ethyl, Ethyl, m.  $155^{\circ}$ .<sup>49</sup>

Phenyl, m.  $175^{\circ}$ .<sup>138</sup>

Propyl, Propyl, m.  $112^{\circ}$ .<sup>49</sup>

*c*-Hexyl, *c*-Hexyl, m.  $261^{\circ}$ .<sup>138</sup> 2-methiodide, m.  $212^{\circ}$ .<sup>135</sup>

Phenyl, Phenyl, m.  $261^{\circ}$ .<sup>138</sup>  $251^{\circ}$ .<sup>135</sup>  $246^{\circ}$ .<sup>260</sup>

## Cyclic 5,5-Disubstituted Dithiohydantoin

-(CH<sub>2</sub>)<sub>4</sub>-, m.  $243^{\circ}$ .<sup>49</sup>

-CH<sub>2</sub>CHMeCH<sub>2</sub>CH<sub>2</sub>-, m.  $232^{\circ}$ .<sup>49</sup>

-(CH<sub>2</sub>)<sub>5</sub>-, m.  $267^{\circ}$ .<sup>49</sup>

-CHMe(CH<sub>2</sub>)<sub>4</sub>-, m.  $184^{\circ}$ .<sup>49</sup>

-CH<sub>2</sub>CHMe(CH<sub>2</sub>)<sub>3</sub>-, m.  $203^{\circ}$ .<sup>49</sup>

-(CH<sub>2</sub>)<sub>2</sub>CHMe(CH<sub>2</sub>)<sub>2</sub>-, m.  $255^{\circ}$ .<sup>49</sup>

-CHMeCH<sub>2</sub>CHMe(CH<sub>2</sub>)<sub>2</sub>-, m.  $172^{\circ}$ .<sup>49</sup>

-CHMe(CH<sub>2</sub>)<sub>2</sub>CHMeCH<sub>2</sub>-, m.  $197^{\circ}$ .<sup>49</sup>

-CH<sub>2</sub>CHMeCH<sub>2</sub>CHMeCH<sub>2</sub>-, m.  $230^{\circ}$ .<sup>49</sup>

-(CH<sub>2</sub>)<sub>6</sub>-, m.  $242^{\circ}$ .<sup>49</sup>

-CH<sub>2</sub>CHMe(CH<sub>2</sub>)<sub>4</sub>-, m.  $220-2^{\circ}$ .<sup>36</sup>

-(CH<sub>2</sub>)<sub>2</sub>CHMe(CH<sub>2</sub>)<sub>3</sub>-, mixt. of stereoisomers, m.  $224^{\circ}$ .<sup>36</sup>

*o*-CH<sub>2</sub>C<sub>6</sub>H<sub>10</sub>-CH<sub>2</sub>CH<sub>2</sub>-, *cis* m.  $242^{\circ}$ .<sup>36</sup>

-heptamethylene spirohydantoin, m.  $263.5^{\circ}$ .<sup>228</sup>

## TRISUBSTITUTED DITHIOHYDANTOIN

1-Methyl-5,5-pentamethylene, m.  $193^{\circ}$ .<sup>52</sup>

1,5-Dimethyl-5-phenyl-, m.  $195^{\circ}$ .<sup>51</sup>

3,5-Dimethyl-5-phenyl-, m. 132°. <sup>51</sup>

1,3-Dibenzyl-5-phenyl-, m. 114°; HCl, m. 168–70°. <sup>67</sup>

#### TETRASUBSTITUTED DITHIOHYDANTOIN

1,3-Dimethyl-5,5-pentamethylene, m. 127°. <sup>52</sup>

1,3-Dimethyl-5,5-diphenyl, m. 167°. <sup>52</sup>

$R_2\overline{C}\cdot NH\cdot \overline{C}(SH):NCS(A)$  and  $R_2\overline{C}\cdot NH\cdot \overline{CS}\cdot N:\overline{C}(SH)(B)$

A) 5,5-Dimethyl,  $Me_2NH$  salt, m. 125°; <sup>135</sup>  $Et_2NH$  salt, m. 125°; <sup>107</sup> Ag salt, m. about 130°; <sup>135</sup> Ca salt, m. 200°, <sup>107</sup> about 200°; Cu salt, m. about 120°; <sup>135</sup> Mg salt, m. 150°; <sup>107</sup> Pb salt, m. about 200°. <sup>135</sup>

$S^2,5,5$ -Trimethyl, m. 136°, <sup>107</sup> 168°; <sup>55, 135</sup> methiodide, m. 195°; <sup>55</sup> 4-methiodide, m. 210°. <sup>107</sup>

$S^2$ -Methyl-5,5-di-*c*-hexyl-, m. 160°. <sup>135</sup>

$S^4,5,5$ -Trimethyl, m. 136°; 2-methiodide, m. 144°. <sup>135</sup>

$S^4$ -Methyl-5,5-di-*c*-hexyl, m. 170°. <sup>135</sup>

$S^4$ -Benzyl-5,5-dimethyl, m. 150°. <sup>135</sup>

$S^2, S^4, 5, 5$ -tetramethyl, m. 35°. <sup>135</sup>

#### Physical Properties of Pseudothiohydantoin and Some of Its Substitution Products

Pseudothiohydantoin, m. 227°, <sup>112</sup> >200°, <sup>203</sup> 200–5°, <sup>257</sup> dec. 250°. <sup>198</sup>

Isothiohydantoin, m. 71°. <sup>333</sup>

#### 2-SUBSTITUTED PSEUDOTHIOHYDANTOINS

Ethyl, m. 145°, <sup>109</sup> 144°; <sup>5</sup> HCl, m. 190–2°; <sup>109</sup> picrate, m. 157°. <sup>5</sup>

*s*-Butyl, HCl, m. 227°. <sup>109</sup>

*i*-Amyl, HCl, m. 195°. <sup>109</sup>

*i*-Hexyl, HCl, m. 180–3°. <sup>109</sup>

Allyl, m. 103°; HCl, m. 177–9°. <sup>109</sup>

$CH_2CCl:CH_2$ , m. 149°; HCl, m. 186°. <sup>84</sup>

*c*-Hexyl, HCl, m. 256°. <sup>109</sup>

Phenyl, m. 179°, <sup>28</sup> 178°, <sup>86, 109</sup> 198, <sup>272, 346, 356</sup> 176–8°, <sup>271</sup> 175°, <sup>195</sup> 176°, <sup>22</sup> 215°; <sup>5</sup> semicarbazone, m. 194°; <sup>22</sup> HCl, m. 215°, <sup>5</sup> 203° dec.; diAc., m. 162°. <sup>356</sup>

Benzyl, m. 188°; HCl, m. 205–7°. <sup>109</sup>

Phenethyl, m. 188°. <sup>348</sup>

$Ph_2CH$ , m. 253°. <sup>309</sup>

- $o$ -Tolyl, m.  $155^{\circ}$ ,<sup>22</sup>  $145^{\circ}$ ; <sup>84, 356</sup> semicarbazone, m.  $182^{\circ}$ ; <sup>22</sup> diAc., m.  $92^{\circ}$ .<sup>356</sup>  
 $p$ -Tolyl, m.  $183^{\circ}$ ,<sup>346</sup>  $182^{\circ}$ ,<sup>109</sup>  $191^{\circ}$ ,<sup>74</sup>  $128^{\circ}$ ; semicarbazone, m.  $152^{\circ}$ ; <sup>22</sup> diAc., m.  $164^{\circ}$ .<sup>356</sup>  
 $\alpha$ -Naphthyl, m.  $184^{\circ}$ ,<sup>73, 195</sup>  $173^{\circ}$ ; semicarbazone, m.  $186^{\circ}$ .<sup>22</sup>  
 $\beta$ -Naphthyl, m.  $225^{\circ}$ ,<sup>234</sup>  $220^{\circ}$ ,<sup>195</sup>  $214^{\circ}$ ,<sup>156</sup>  $193^{\circ}$ ; semicarbazone, m.  $208^{\circ}$ .<sup>22</sup>  
 $\text{Me}_2\text{C}_6\text{H}_3$ , 2,4-, m.  $157^{\circ}$ ; <sup>156</sup> 3,4-, m.  $179^{\circ}$ ; 2,5-, m.  $162^{\circ}$ .<sup>356</sup>  
 $\text{MeOC}_6\text{H}_4$ ,  $o$ , m.  $136^{\circ}$ ;  $m$ , m.  $160^{\circ}$ ;  $p$ , m.  $191^{\circ}$ ,<sup>195</sup>  $108^{\circ}$ ; semicarbazone, m.  $120^{\circ}$ .<sup>22</sup>  
 $p$ -EtOC<sub>6</sub>H<sub>4</sub>, m.  $166^{\circ}$ ,<sup>110</sup>  $164^{\circ}$ ,<sup>356</sup>  $106^{\circ}$ ; semicarbazone, m.  $140^{\circ}$ .<sup>22</sup>  
 $p$ -PrOC<sub>6</sub>H<sub>4</sub>, m.  $168^{\circ}$ .<sup>110</sup>  
 $\text{BuOC}_6\text{H}_4$ ,  $m$ , m.  $137^{\circ}$ ;  $p$ , m.  $160^{\circ}$ .<sup>110</sup>  
 $\text{ClC}_6\text{H}_4$ ,  $o$ , m.  $144^{\circ}$ ;  $m$ , m.  $181^{\circ}$ ,<sup>195</sup>  $180^{\circ}$ ; <sup>109</sup>  $p$ , m.  $200^{\circ}$ ; <sup>195</sup>  $\text{Br}_2$  addn. compd., m.  $294^{\circ}$ .<sup>222</sup>  
 $\text{O}_2\text{NC}_6\text{H}_4$ ,  $o$ , m.  $147^{\circ}$ ;  $m$ , m.  $210^{\circ}$ ;  $p$ , m.  $235^{\circ}$ ,<sup>195</sup>  $225^{\circ}$ .<sup>233</sup>  
 $p$ -H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>, m.  $206^{\circ}$ .<sup>233</sup>  
 $\text{HOCC}_6\text{H}_4$ ,  $o$ , m.  $190^{\circ}$ ;  $m$ , m.  $255^{\circ}$ ;  $p$ , m.  $>360^{\circ}$ .<sup>195</sup>  
2,4-AcOHg(Cl)C<sub>6</sub>H<sub>3</sub>, m.  $215^{\circ}$  dec.<sup>222</sup>  
2,4-AcOHg(NH<sub>2</sub>)C<sub>6</sub>H<sub>3</sub>, m.  $207^{\circ}$ .<sup>233</sup>  
 $\text{Me}_2\text{C}_6\text{H}_4\text{CH}_2$ , (cuminy), m.  $225$ – $35^{\circ}$  dec.<sup>330</sup>  
 $\alpha$ -( $\beta$ -AcOHgC<sub>10</sub>H<sub>6</sub>), m.  $205^{\circ}$ .<sup>73</sup>



| XHgR'  | AcO            | Cl                  | Br                  |
|--|----------------|---------------------|---------------------|
|  | m.             | m.                  | m.                  |
| $p$ -XHgC <sub>6</sub> H <sub>4</sub> -                  | $220^{\circ}$  | $345^{\circ}$ dec   | $280^{\circ}$       |
| 2,4-Me(XHg)C <sub>6</sub> H <sub>3</sub> -,              | $185^{\circ}$  | $315^{\circ}$       | $>350^{\circ}$      |
| 3,4-Me(XHg)C <sub>6</sub> H <sub>3</sub> -,              | $192^{\circ}$  | $350^{\circ}$ dec.  | $276^{\circ}$       |
| 2,4-XHg(Me)C <sub>6</sub> H <sub>3</sub> ,               | $211^{\circ}$  | $260^{\circ}$       | $>350^{\circ}$      |
| 2,4-O <sub>2</sub> N(XHg)C <sub>6</sub> H <sub>3</sub> , | $220^{\circ}$  | $195^{\circ}$       | $>350^{\circ}$      |
| 3,4-O <sub>2</sub> N(XHg)C <sub>6</sub> H <sub>3</sub> , | $190^{\circ}$  | $212^{\circ}$       | $165^{\circ}$       |
| 2,4-XHg(O <sub>2</sub> N)C <sub>6</sub> H <sub>3</sub> , | $206^{\circ}$  | $242^{\circ}$       | $198^{\circ}$       |
| 2,4-Cl(XHg)C <sub>6</sub> H <sub>3</sub> ,               | $207^{\circ}$  | $>350^{\circ}$ dec. | $180^{\circ}$       |
| 3,4-Cl(XHg)C <sub>6</sub> H <sub>3</sub> ,               | $178^{\circ}$  | $180^{\circ}$       | $260^{\circ}$ dec.  |
| 2,4-XHg(Cl)C <sub>6</sub> H <sub>3</sub> ,               | $190^{\circ}$  | $170^{\circ}$       | $>350^{\circ}$ dec. |
| 2,4-HOOC(XHg)C <sub>6</sub> H <sub>3</sub> ,             | $240^{\circ}$  | $345^{\circ}$       | $>350^{\circ}$ dec. |
| 3,4-HOOC(XHg)C <sub>6</sub> H <sub>3</sub> ,             | $263^{\circ}$  | $280^{\circ}$       | $278^{\circ}$ dec.  |
| 2,4-XHg(HOOC)C <sub>6</sub> H <sub>3</sub> ,             | $>350^{\circ}$ | $290^{\circ}$ dec.  | $290^{\circ}$ dec.  |

## 3-SUBSTITUTED PSEUDOTHIOHYDANTOIN

Butyl, m.  $103^{\circ}$ .<sup>242</sup>  
 Hexyl, m.  $78^{\circ}$ .<sup>242</sup>  
 Decyl, m.  $54^{\circ}$ .<sup>242</sup>  
*c*-Hexyl, m.  $123-5^{\circ}$ .<sup>242</sup>  
 Allyl, HCl, m.  $176^{\circ}$ .<sup>94</sup>  
 Phenyl, m.  $148^{\circ}$ ,<sup>356</sup>  $142^{\circ}$ ; <sup>331</sup> picrate, m.  $180^{\circ}$ .<sup>331</sup>  
 Tollyl, *o*, m.  $132^{\circ}$ ; <sup>356</sup> *m*, m.  $161^{\circ}$ ; <sup>320</sup> *p*, m.  $126^{\circ}$ .<sup>332</sup>  
 $\text{Me}_2\text{C}_6\text{H}_3$ , 2,5-, m.  $110^{\circ}$ .<sup>356</sup>  
 $\text{Me}_3\text{C}_6\text{H}_2$ , 2,4,5-, m.  $210^{\circ}$ .<sup>320</sup>  
 Naphthyl,  $\beta$ , m.  $147^{\circ}$ .<sup>156</sup>  
 $\text{O}_2\text{NC}_6\text{H}_4$ , *o*, m.  $172^{\circ}$ ; *m*, m.  $199^{\circ}$ ,<sup>320</sup>  $184^{\circ}$ ; <sup>156</sup> *p*, m.  $245^{\circ}$ .<sup>320</sup>  
 $\text{Me}(\text{O}_2\text{N})\text{C}_6\text{H}_3$ , 2,4-, m.  $172^{\circ}$ ; 4,2-, m.  $185^{\circ}$ ; 4,3-, m.  $207^{\circ}$ .<sup>320</sup>  
 $\text{MeOC}_6\text{H}_4$ , *o*, m.  $149^{\circ}$ ; *p*, m.  $184^{\circ}$ .<sup>320</sup>  
 $\text{EtOC}_6\text{H}_4$ , *p*, m.  $167^{\circ}$ ,<sup>320</sup>  $165^{\circ}$ .<sup>332</sup>  
 $\text{PhOC}_6\text{H}_4$ , *o*, m.  $97.5^{\circ}$ ; *p*, m.  $133^{\circ}$ .<sup>231</sup>  
 $\text{ClC}_6\text{H}_4$ , *m*, m.  $181^{\circ}$ ; *p*, m.  $213^{\circ}$ .<sup>320</sup>  
 $p\text{-H}_2\text{NSO}_2\text{C}_6\text{H}_4$ , m.  $258^{\circ}$ .<sup>270</sup>  
 $\text{HOOC}$ , Et ester, m.  $174^{\circ}$ ; *i*-Am ester, m.  $147^{\circ}$ .<sup>327</sup>  
 $\text{PhNH}$ , m.  $176^{\circ}$ ; <sup>329</sup>, <sup>334</sup> HCl, m.  $190^{\circ}$ .<sup>334</sup>  
 $p\text{-H}_2\text{NC}_6\text{H}_4\text{SO}_2$ , m.  $237^{\circ}$  dec.; Ac., m.  $259^{\circ}$  dec.<sup>272</sup>  
 $\text{Me}_2\text{C:N}$ , m.  $176^{\circ}$ .<sup>272</sup>  
 $\text{PhCH:N}$ , m.  $255^{\circ}$ .<sup>272</sup>  
 $\text{PhCH}_2\text{CH:N}$ , m.  $164^{\circ}$  dec.<sup>272</sup>

## 5-SUBSTITUTED PSEUDOTHIOHYDANTOIN

Methyl, m.  $226^{\circ}$ ,<sup>353</sup>  $206^{\circ}$  dec.,<sup>84</sup>  $205^{\circ}$  dec.<sup>5</sup>  
 Ethyl, m.  $200^{\circ}$ ,<sup>5</sup>  $196-8^{\circ}$ .<sup>244</sup>  
 Heptyl, m.  $191.5^{\circ}$ .<sup>244</sup>  
 Nonyl, m.  $182.5^{\circ}$ .<sup>244</sup>  
 Cetyl, m.  $174.5^{\circ}$ .<sup>244</sup>  
 Allyl, m.  $180^{\circ}$ .<sup>220</sup>  
 Phenyl, m.  $257^{\circ}$ .<sup>353</sup>  
 $\text{EtOOC}$ , m. about  $175^{\circ}$ .<sup>92</sup>  
 $\text{HOOCCH}_2$ , m.  $210-12^{\circ}$ ; <sup>157</sup>  $\text{C}_{14}\text{H}_{29}$  ester, m.  $66^{\circ}$ ; octadecenyl  
 ester, m.  $225^{\circ}$ .<sup>315</sup>  
 $\text{HOOCCHMe}$ , m.  $225^{\circ}$ .<sup>5</sup>  
 $\text{HON:}$ , Electrolytic dissociation,  $K_{25} 5.5 \times 10^{-8}$ .<sup>5</sup>  
 1-HO-*c*-hex, m.  $245^{\circ}$ .<sup>109</sup>

Tolylazo, m. 192°. <sup>24</sup>

*p*-Sulfonamidophenylazo, m. 225°. <sup>24</sup>

### 5,5'-bis-PSEUDOTHIOHYDANTOINS

Ethylene, m. 270°. <sup>79</sup>

Tetramethylene, dec. 260–2°. <sup>215</sup>

Hexamethylene, m. 249–51° dec. <sup>79</sup>

### 2,3-DISUBSTITUTED PSEUDOTHIOHYDANTOINS

2            3

Methyl, Methyl, m. 71°. <sup>5</sup>

Ethyl, m. 44°. <sup>5</sup> (May be 2-Et, 3-Me.)

Allyl, oil. <sup>5</sup> (May be 2-Allyl, 3-Me.)

Ethyl, Ethyl, m. 41°, <sup>5</sup> 38°. <sup>294</sup>

Allyl, oil. <sup>5</sup>

Phenyl, oil. <sup>5</sup>

Tolyl, *m*, m. 107°. <sup>320</sup>

Me<sub>3</sub>C<sub>6</sub>H<sub>2</sub>, 2,4,5-, m. 138°. <sup>320</sup>

O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>, *p*, m. 129°. <sup>320</sup>

O<sub>2</sub>N(Me)C<sub>6</sub>H<sub>3</sub>, 4,2-, m. 128°; 3,4-, m. 142°. <sup>320</sup>

ClC<sub>6</sub>H<sub>4</sub>, *p*, m. 107°. <sup>320</sup>

MeOC<sub>6</sub>H<sub>4</sub>, *o*, m. 127°; *p*, m. 85°. <sup>320</sup>

EtOC<sub>6</sub>H<sub>4</sub>, *p*, m. 95°. <sup>320</sup>

Allyl, Allyl, b<sub>0.13</sub> 85°; d 20/4 1.1421; n 20/D 1.5548. <sup>109</sup>

*i*-Amyl, *i*-Amyl, b<sub>0.1</sub> 109°; d 20/4 1.3073; n 20/D 1.5022. <sup>109</sup>

Phenyl, Cetyl, m. 56°. <sup>243</sup>

Phenyl, m. 176°, <sup>21</sup> 22, <sup>272</sup> 343 177°; 3, <sup>86</sup> semicarbazone,  
m. 194°. <sup>22</sup>

Benzyl, m. 82°. <sup>242</sup>

Benzyl, Benzyl, m. 84–6°. <sup>242</sup>

Phenethyl, Phenethyl, m. 25–8°. <sup>242</sup>

Ph<sub>2</sub>CH, Methyl, m. 246°. <sup>309</sup>

*o*-Tolyl, *o*-Tolyl, m. 152°, <sup>26</sup> 355 155°, <sup>22</sup> 153°; <sup>323</sup> semicarbazone,  
m. 156°; phenyl carbazone, m. 155°; <sup>26</sup> 2,4-dinitrophenyl-  
hydrazone, m. 158°; 1,1-dioxide, m. 134°. <sup>26</sup>

*m*-Tolyl, *m*-Tolyl, m. 154°; HCl, m. 180–2°; 1,1-dioxide, m.  
214°. <sup>25</sup>

*p*-Tolyl, *p*-Tolyl, m. 128°, <sup>22</sup> 127°; HCl, m. 195°; 1,1-dioxide, m.  
147°; <sup>24</sup> semicarbazone, m. 152°. <sup>22</sup>

*p*-HOC<sub>6</sub>H<sub>4</sub>, *p*-HOC<sub>6</sub>H<sub>4</sub>, m. 236°. <sup>264</sup>

- 2            3  
 MeOC<sub>6</sub>H<sub>4</sub>, MeOC<sub>6</sub>H<sub>4</sub>, *o,o*, m. 190°,<sup>325</sup> 197°; <sup>226</sup> *p,p*, m. 108°; semicarbazone, m. 120°.<sup>22</sup>  
*p*-EtOC<sub>6</sub>H<sub>4</sub>, *p*-EtOC<sub>6</sub>H<sub>4</sub>, m. 106°; semicarbazone, m. 140°.<sup>22</sup>  
 PrOC<sub>6</sub>H<sub>4</sub>, PrOC<sub>6</sub>H<sub>4</sub>, *p,p*, m. 96–8°.<sup>204</sup>  
*p*-PhOC<sub>6</sub>H<sub>4</sub>, PhCH<sub>2</sub>, m. 108°.<sup>231</sup>  
    *p*-PhOC<sub>6</sub>H<sub>4</sub>, m. 131°.<sup>231</sup>  
 α-C<sub>10</sub>H<sub>7</sub>, α-C<sub>10</sub>H<sub>7</sub>, m. 176°,<sup>324</sup> 197°,<sup>23</sup> 173°; semicarbazone, m. 186°.<sup>22</sup>  
 β-C<sub>10</sub>H<sub>7</sub>, β-C<sub>10</sub>H<sub>7</sub>, m. 174°,<sup>324</sup> 192°,<sup>23</sup> 193°; semicarbazone, m. 208°.<sup>22</sup>  
 3,3'-Diethyl-2,2'-bis-pseudothiohydantoin, m. 224°.<sup>115</sup>  
 2,2'-Ethylene-bis-3-ethyl-pseudothiohydantoin, m. 184°.<sup>347</sup>  
 2,4-Me<sub>2</sub>C<sub>6</sub>H<sub>3</sub>, Acetyl, m. 166°.<sup>156</sup>  
 β-C<sub>10</sub>H<sub>7</sub>, Acetyl, m. 143°.<sup>156</sup>  
*p*-MeOC<sub>6</sub>H<sub>4</sub>, *p*-AcNHC<sub>6</sub>H<sub>4</sub>SO<sub>2</sub>, m. 255° dec.<sup>272</sup>  
    PhCH<sub>2</sub>CH:N, m. 220° dec.<sup>272</sup>  
 Acetyl, Phenyl, m. 192°.<sup>356</sup>  
    *p*-Tolyl, m. 176°.<sup>356</sup>  
    *p*-EtOC<sub>6</sub>H<sub>4</sub>, m. 155°.<sup>356</sup>  
    β-C<sub>10</sub>H<sub>7</sub>, m. 140°.<sup>156</sup>  
 Benzoyl, *p*-BrC<sub>6</sub>H<sub>4</sub>, m. 213°.<sup>94</sup>  
    PhOC<sub>6</sub>H<sub>4</sub>, *o*, m. 164°; *p*, m. 214°.<sup>231</sup>

## 2,5-DISUBSTITUTED PSEUDOTHIOHYDANTOINS

- 2            5  
 Ethyl, Methyl, oil.<sup>5</sup>  
 Phenyl, Methyl, m. 239°.<sup>86</sup>  
    Ethyl, m. 149°.<sup>84</sup>  
    Phenyl, m. 186°,<sup>354</sup> 185°.<sup>94</sup>  
    CHMeCOOH, m. 214°.<sup>5</sup>  
    CO·COOH, m. 222° dec.<sup>355</sup>  
*o*-Tolyl, Methyl, m. 73°.<sup>84</sup>  
    Ethyl, m. 95–8°; HBr, m. 225°.<sup>84</sup>  
*p*-Tolyl, Sulfanilamidophenyl, m. 185°.<sup>219</sup>  
    Benzyl, m. 119°.<sup>74</sup>  
    HOC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>, *o*, m. 160° dec.; *p*, m. 104°.<sup>74</sup>  
    *p*-MeOC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>, m. 186°.<sup>74</sup>  
    4,3-HO(MeO)C<sub>6</sub>H<sub>3</sub>CH<sub>2</sub>, m. 126°.<sup>74</sup>

2 5

5,4,3-Br (HO) (MeO)C<sub>6</sub>H<sub>2</sub>CH<sub>2</sub>, m. 95°. <sup>74</sup>*p*-Me<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>, m. 280°. <sup>74</sup>PhCH:CHCH<sub>2</sub>, m. 113°. <sup>74</sup>MeOC<sub>6</sub>H<sub>4</sub>, *p*-Sulfamoylphenylazo, *o*, m. 102°; *m*, m. >350°; *p*, m. 285°. <sup>195</sup>O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>, *p*-Sulfamoylphenylazo, *o*, m. 70°; *m*, m. 112°. <sup>195</sup>*p*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>, Phenylazo, m. >300°. <sup>233</sup>*p*-Sulfamoylphenylazo, m. 78°. <sup>195</sup>-NHC(:NH)NHC(:NH)NHR, Ph, m. 245°; *p*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>, m. 164°; *p*-ClC<sub>6</sub>H<sub>4</sub>, m. 164°; *p*-BrC<sub>6</sub>H<sub>4</sub>, m. 178°; β-C<sub>10</sub>H<sub>7</sub>, m. 216°. <sup>233</sup>ClC<sub>6</sub>H<sub>4</sub>, *p*-Sulfamoylphenylazo, *o*, m. 118°; *m*, m. 195°; *p*, m. 150°. <sup>195</sup>HOCC<sub>6</sub>H<sub>4</sub>, *p*-Sulfamoylphenylazo, *o*, m. >350°; *m*, m. 350°; *p*, m. >350°. <sup>195</sup>C<sub>10</sub>H<sub>7</sub>, *p*-Sulfamoylphenylazo, α, m. 105°; β, m. 135°. <sup>195</sup>

## 5,5-DISUBSTITUTED PSEUDOTHIOHYDANTOINS

Methyl, Methyl, m. 265°, <sup>269</sup> 242°. <sup>5</sup>Ethyl, m. 242°. <sup>269</sup>*i*-Butyl, m. 247°. <sup>269</sup>Hexyl, m. 218°. <sup>269</sup>CH<sub>2</sub>COOH, m. 235°. <sup>109</sup>Ethyl, Ethyl, m. 225°, <sup>253</sup> 224°. <sup>60</sup>Butyl, m. 203°. <sup>253</sup>Phenyl, m. 213°. <sup>253</sup>Propyl, Propyl, m. 235°. <sup>253</sup>Butyl, Butyl, m. 223°. <sup>253</sup>Phenyl, Phenyl, m. 274-6°, <sup>72</sup> 270°. <sup>235</sup>

## 2,3,5-TRISUBSTITUTED PSEUDOTHIOHYDANTOINS

Methyl, Methyl, HON: m. 220°. <sup>5</sup>Ethyl, Phenyl, Methyl, m. 101°. <sup>5</sup>Phenyl, Phenyl, HOCCCH<sub>2</sub>, m. 189.5°, <sup>352</sup> 188°; <sup>5</sup> HCl, m. 193-5°. <sup>352</sup>HOCCCHMe, m. 124°. <sup>5</sup>*m*-Tolyl, *m*-Tolyl, *p*-Sulfonamidophenylazo, m. 235° dec. <sup>25</sup>*o*-Anisyl, *o*-Anisyl, *p*-Sulfonamidophenylazo, m. 183°. <sup>226</sup>



*o*-Phenetyl, *o*-Phenetyl, *p*-Sulfonamidophenylazo, m. 145–50°. <sup>226</sup>  
 $\text{ClC}_6\text{H}_4$ ,  $\text{ClC}_6\text{H}_4$ , *o,o*, m. 168°; *m,m*, m. 125°; *p,p*, m. 156–8°. <sup>226</sup>  
 $\text{C}_{10}\text{H}_7$ ,  $\text{C}_{10}\text{H}_7$ , *p*-Tolueneazo,  $\alpha$ , m. 235°;  $\beta$ , m. 104°. <sup>23</sup>

### 2,5,5-TRISUBSTITUTED PSEUDOTHIOHYDANTOINS

Triphenyl, m. 250°. <sup>356</sup>  
 $\text{Ph}_2\text{CH}$ , Phenyl, Phenyl, m. 328° dec. <sup>72</sup>  
 $\beta$ - $\text{C}_{10}\text{H}_7$ , Br,  $\text{CHBrAr}$ , <sup>234</sup>  $\text{Ar}=\text{furyl}$ , m. >360°; Ph, m. 228°;  
 $\text{HOC}_6\text{H}_4$ , *o*, m. 120°; *p*, m. 208°; *p*- $\text{MeOC}_6\text{H}_4$ , m. 103°; 4,3-  
 $\text{HO}(\text{MeO})\text{C}_6\text{H}_3$ , m. 155°;  $\text{O}_2\text{NC}_6\text{H}_4$ , *o*, m. 91°; *m*, m. 161°;  
*p*, m. 295°; *p*- $\text{Me}_2\text{NC}_6\text{H}_4$ , m. 156°;  $\text{CHBrCBr:CBPh}$ , m. 128°.

### SELENO COMPOUNDS

Pseudoselenohydantoin, m. 190° dec. <sup>327, 335</sup>  
5-Me-pseudoselenohydantoin, m. 179°. <sup>327</sup>

### 5-ALDEHYDO COMPOUNDS OF PSEUDOTHIOHYDANTOIN

#### Aldehyde or Ketone

*c*-Pentanone, dec. 225–30°. <sup>109</sup>  
*c*-Hexanone, m. 252°. <sup>109</sup>  
 $\text{PhCH:CHCHO}$ , dec. 235°. <sup>357</sup>  
*o*- $\text{HOC}_6\text{H}_4\text{CHO}$ , m. 215°; Ac., m. 223–8° dec. <sup>357</sup>  
3,4-( $\text{MeO}$ ) $_2\text{C}_6\text{H}_3\text{CHO}$ , dec. 255°. <sup>192</sup>  
*m*- $\text{O}_2\text{NC}_6\text{H}_4\text{CHO}$ , m. 260°, <sup>264</sup> dec. 268°. <sup>314</sup>  
*p*- $\text{AcNHC}_6\text{H}_4\text{CHO}$ , m. 314° dec., <sup>314</sup> 325°. <sup>270</sup>  
*p*- $\text{Me}_2\text{NC}_6\text{H}_4\text{CHO}$ , m. 236–8°. <sup>314</sup>

### 2-SUBSTITUTED-5-ALDEHYDO-PSEUDOTHIOHYDANTOIN

Allyl,  $\text{PhCHO}$ , m. 165°. <sup>109</sup>  
Phenyl,  $\text{HCHO}$ , m. 250°. <sup>73</sup>  
 $\text{PhCHO}$ , m. 254°, <sup>323</sup> 253°, <sup>356</sup> 201°. <sup>5</sup>  
*p*- $\text{MeC}_6\text{H}_4\text{CHO}$ , m. 261°. <sup>73</sup>  
 $\text{HOC}_6\text{H}_4\text{CHO}$ , *o*, m. 192°; *m*, m. 102°; *p*, m. 120°. <sup>28</sup>  
*p*- $\text{MeOC}_6\text{H}_4\text{CHO}$ , m. 94°. <sup>28</sup>  
3,4- $\text{CH}_2\text{O}_2\text{C}_6\text{H}_3\text{CHO}$ , m. 147°. <sup>28</sup>  
 $\text{O}_2\text{NC}_6\text{H}_4\text{CHO}$ , *o*, m. 205°; *m*, m. 115°; *p*, m. 275°. <sup>73</sup>  
 $\text{ClC}_6\text{H}_4\text{CHO}$ , *o*, m. 184°, <sup>28</sup> 150°; *m*, m. 204–6°; *p*, m.  
260°, <sup>73</sup> 132°. <sup>28</sup>  
 $\text{C}_{10}\text{H}_7\text{CHO}$ ,  $\alpha$ , m. 165°;  $\beta$ , m. 227°. <sup>73</sup>  
 $\text{PhN:CHCHO}$ , m. 202°. <sup>323</sup>

- Benzyl,  $\text{PhCHO}$ , m.  $218^\circ$ .<sup>109</sup>
- p*-Tolyl,  $2\text{-C}_4\text{H}_3\text{O}\cdot\text{CHO}$ , m.  $234^\circ$ .<sup>74</sup>
- $\text{PhCHO}$ , m.  $268^\circ$ .<sup>74</sup>
- $\text{PhCH:CHCHO}$ , m.  $222^\circ$ .<sup>74</sup>
- $\text{HOC}_6\text{H}_4\text{CHO}$ , *o*, m.  $253^\circ$ ; *p*, m.  $298^\circ$ .<sup>74</sup>
- $p\text{-MeOC}_6\text{H}_4\text{CHO}$ , m.  $250^\circ$ .<sup>74</sup>
- $\text{O}_2\text{NC}_6\text{H}_4\text{CHO}$ , *o*, m.  $260^\circ$ ; *m*, m.  $285^\circ$ ; *p*, m.  $316^\circ$ .<sup>74</sup>
- $\text{HO}(\text{O}_2\text{N})\text{C}_6\text{H}_3\text{CHO}$ , 2,3-, m.  $267^\circ$ ; 2,5-, m.  $283^\circ$ ; 4,3-, m.  $220^\circ$ .<sup>74</sup>
- 4,3-MeO( $\text{O}_2\text{N}$ ) $\text{C}_6\text{H}_3\text{CHO}$ , m.  $250^\circ$ .<sup>74</sup>
- 5,4,3-Br( $\text{HO}$ )(MeO) $\text{C}_6\text{H}_2\text{CHO}$ , m.  $216^\circ$ .<sup>74</sup>
- $p\text{-Me}_2\text{NC}_6\text{H}_4\text{CHO}$ , m.  $294^\circ$ .<sup>74</sup>
- p*- $\text{O}_2\text{NC}_6\text{H}_4\text{-}$ ,  $\text{PhCHO}$ , m.  $311^\circ$ .<sup>233</sup>
- $\text{PhCH:CHCHO}$ , m.  $265^\circ$ .<sup>233</sup>
- $\text{HOC}_6\text{H}_4\text{CHO}$ , *o*, m.  $252^\circ$ ; *p*, m.  $>300^\circ$ .<sup>233</sup>
- $p\text{-MeOC}_6\text{H}_4\text{CHO}$ , m.  $320^\circ$ .<sup>233</sup>
- Vanillin, m.  $256^\circ$ .<sup>233</sup>
- $\text{O}_2\text{NC}_6\text{H}_4\text{CHO}$ , *o*, m.  $264^\circ$ ; *m*, m.  $247^\circ$ ; *p*, m.  $301^\circ$ .<sup>233</sup>
- p*- $\text{ClC}_6\text{H}_4\text{-}$ ,  $2\text{-C}_4\text{H}_3\text{O}\cdot\text{CHO}$ , m.  $>260^\circ$ .<sup>222</sup>
- $\text{PhCHO}$ , m.  $>260^\circ$ ; <sup>222</sup> dibromide, m.  $280^\circ$ .<sup>222</sup>
- $\text{PhCH:CHCHO}$ , m.  $220^\circ$ .<sup>222</sup>
- $\text{HOC}_6\text{H}_4\text{CHO}$ , *o*, m.  $>300^\circ$ ; *p*, m.  $>250^\circ$ .<sup>222</sup>
- $p\text{-MeOC}_6\text{H}_4\text{CHO}$ , m.  $>260^\circ$ .<sup>222</sup>
- 3,4-MeO( $\text{HO}$ ) $\text{C}_6\text{H}_3\text{CHO}$ , m.  $210^\circ$ .<sup>222</sup>
- $\text{O}_2\text{NC}_6\text{H}_4\text{CHO}$ , *o*, m.  $>250^\circ$ ; *m*, m.  $251^\circ$ ; *p*, m.  $235^\circ$ .<sup>222</sup>
- 2,5- $\text{HO}(\text{O}_2\text{N})\text{C}_6\text{H}_3\text{CHO}$ , m.  $>250^\circ$ .<sup>222</sup>
- $p\text{-Me}_2\text{NC}_6\text{H}_4\text{CHO}$ , m.  $>260^\circ$ .<sup>222</sup>
- p*- $\text{H}_2\text{NC}_6\text{H}_4\text{-}$ ,  $\text{PhCHO}$ , m.  $320^\circ$ .<sup>233</sup>
- $\text{PhCH:CHCHO}$ , m.  $167^\circ$ .<sup>233</sup>
- $\text{HOC}_6\text{H}_4\text{CHO}$ , *o*, m.  $180^\circ$ ; *p*, m.  $>300^\circ$ .<sup>233</sup>
- $p\text{-MeOC}_6\text{H}_4\text{CHO}$ , m.  $280^\circ$  dec.<sup>233</sup>
- Vanillin, m.  $255^\circ$ .<sup>233</sup>
- $\text{O}_2\text{NC}_6\text{H}_4\text{CHO}$ , *o*, m.  $290^\circ$  dec.; *m*, m.  $>300^\circ$ ; *p*, m.  $>300^\circ$ .<sup>233</sup>
- p*- $\text{Me}_2\text{NC}_6\text{H}_4\text{-}$ , *p*- $\text{O}_2\text{NC}_6\text{H}_4\text{CHO}$ , m.  $250\text{--}52^\circ$ .<sup>356</sup>
- Naphthyl,  $2\text{-C}_4\text{H}_3\text{O}\cdot\text{CHO}$ ,  $\alpha$ , m.  $210^\circ$  dec.; <sup>73</sup>  $\beta$ , m.  $315^\circ$ .<sup>234</sup>
- $\text{PhCHO}$ ,  $\alpha$ , m.  $184^\circ$ ; <sup>73</sup>  $\beta$ , m.  $321^\circ$ ; <sup>234</sup>  $272^\circ$  dec.<sup>156</sup>
- $\text{PhCH:CHCHO}$ ,  $\alpha$ , m.  $160^\circ$ ; <sup>73</sup>  $\beta$ , m.  $260^\circ$ .<sup>234</sup>

- $o$ -HOC<sub>6</sub>H<sub>4</sub>CHO,  $\alpha$ , m. 155° dec.; <sup>73</sup>  $\beta$ , m. 246°. <sup>234</sup>  
 $p$ -HOC<sub>6</sub>H<sub>4</sub>CHO,  $\alpha$ , m. 120°; <sup>73</sup>  $\beta$ , m. 288°. <sup>234</sup>  
 $p$ -MeOC<sub>6</sub>H<sub>4</sub>CHO,  $\alpha$ , m. 206°; <sup>73</sup>  $\beta$ , m. 331°. <sup>234</sup>  
 3,4-MeO(HO)C<sub>6</sub>H<sub>3</sub>CHO,  $\beta$ , m. 285°. <sup>234</sup>  
 Vanillin,  $\alpha$ , m. 205°. <sup>73</sup>  
 $o$ -O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO,  $\alpha$ , m. 164°; <sup>73</sup>  $\beta$ , m. 240°. <sup>234</sup>  
 $m$ -O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO,  $\alpha$ , m. 196°; <sup>73</sup>  $\beta$ , m. 301°. <sup>234</sup>  
 $p$ -O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO,  $\alpha$ , m. 195°; <sup>73</sup>  $\beta$ , m. 325°. <sup>234</sup>  
 $p$ -Me<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO,  $\alpha$ , m. 180° dec.; <sup>73</sup>  $\beta$ , m. 304°. <sup>234</sup>  
 Michler's ketone,  $\alpha$ , m. 110° dec. <sup>73</sup>  
 Anthraquinone,  $\alpha$ , m. 135°. <sup>73</sup>  
 Alizarin,  $\alpha$ , m. 80°. <sup>73</sup>  
 Isatin, m. 215°. <sup>73</sup>  
 2,4-AcOHg(Cl)C<sub>6</sub>H<sub>3</sub>-, 2-C<sub>4</sub>H<sub>3</sub>O·CHO, m. >300°. <sup>222</sup>  
     PhCHO, m. 225°. <sup>222</sup>  
     PhCH:CHCHO, m. 300°. <sup>222</sup>  
      $p$ -MeC<sub>6</sub>H<sub>4</sub>CHO, m. 245°. <sup>222</sup>  
     HOC<sub>6</sub>H<sub>4</sub>CHO,  $o$ , m. 265°;  $p$ , m. 262°. <sup>222</sup>  
     4,3-HO(MeO)C<sub>6</sub>H<sub>3</sub>CHO, m. 226°. <sup>222</sup>  
     O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO,  $o$ , m. 216°;  $m$ , m. 213°;  $p$ ,  
         m. 235°. <sup>222</sup>  
     2,5-HO(O<sub>2</sub>N)C<sub>6</sub>H<sub>3</sub>CHO, m. 225° dec. <sup>222</sup>  
      $p$ -Me<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO, m. 260°. <sup>222</sup>  
 2,4-AcOHg(H<sub>2</sub>N)C<sub>6</sub>H<sub>3</sub>-, PhCHO, m. 205°. <sup>233</sup>  
     PhCH:CHCHO, m. 230°. <sup>233</sup>  
      $p$ -MeOC<sub>6</sub>H<sub>4</sub>CHO, m. 235°. <sup>233</sup>  
      $p$ -O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO, m. 205°. <sup>233</sup>  
 2,1-AcOHgC<sub>10</sub>H<sub>6</sub>-, PhCHO, m. 210°. <sup>73</sup>

### 3-SUBSTITUTED-5-ALDEHYDO-PSEUDOTHIOHYDANTOIN

- Phenyl, PhCHO, m. 256°. <sup>5</sup>  
      $o$ -HOC<sub>6</sub>H<sub>4</sub>CHO, m. 244°. <sup>5</sup>  
 $p$ -PhOC<sub>6</sub>H<sub>4</sub>-, PhCHO, m. 174°. <sup>231</sup>

### 3,2-DISUBSTITUTED-5-ALDEHYDO-PSEUDOTHIOHYDANTOIN

- 3          2  
 Methyl-, Ethyl, (or 2-Me, 3-Et) PhCHO, m. 89°. <sup>5</sup>  
     Allyl, (or 2-Me, 3-Allyl) PhCHO, m. 78°. <sup>5</sup>  
     Ph<sub>2</sub>CH, PhCHO, m. 256–8°. <sup>309</sup>  
      $p$ -Tolyl, 2-C<sub>4</sub>H<sub>3</sub>O·CHO, m. 192°. <sup>74</sup>

3

2

- PhCH:CHCHO, m. 108°. <sup>74</sup>  
 HOC<sub>6</sub>H<sub>4</sub>CHO, *o*, m. 224°; *p*, m. 204°. <sup>74</sup>  
*p*-MeOC<sub>6</sub>H<sub>4</sub>CHO, m. 217°. <sup>74</sup>  
 4,3-HO (MeO)C<sub>6</sub>H<sub>3</sub>CHO, m. 101°. <sup>74</sup>  
 O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO, *o*, m. 108°; *m*, m. 278°; *p*, m.  
 > 325°. <sup>74</sup>  
 HO(O<sub>2</sub>N)C<sub>6</sub>H<sub>3</sub>CHO, 2,3-, m. 246°; 2,5-, m.  
 242°. <sup>74</sup>  
 4,3-MeO (O<sub>2</sub>N)C<sub>6</sub>H<sub>3</sub>CHO, m. 200°. <sup>74</sup>  
 5,4,3-Br (HO) (MeO)C<sub>6</sub>H<sub>2</sub>CHO, m. 156°. <sup>74</sup>  
*p*-Me<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO, m. 144°. <sup>74</sup>  
*p*-ClC<sub>6</sub>H<sub>4</sub>-, 2-C<sub>4</sub>H<sub>9</sub>O·CHO, m. 174°. <sup>222</sup>  
 PhCHO, m. 79°. <sup>222</sup>  
 PhCH:CHCHO, m. 90°. <sup>222</sup>  
 HOC<sub>6</sub>H<sub>4</sub>CHO, *o*, m. 210°; *p*, m. 85°. <sup>222</sup>  
 3,4-MeO (HO)C<sub>6</sub>H<sub>3</sub>CHO, m. 80°. <sup>222</sup>  
 O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO, *o*, m. 215°; *m*, m. 172°; *p*,  
 m. 251°. <sup>222</sup>  
 5,2-O<sub>2</sub>N (HO)C<sub>6</sub>H<sub>3</sub>CHO, m. 212°. <sup>222</sup>  
*p*-Me<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO, m. 120°. <sup>222</sup>  
 α-Naphthyl, 2-C<sub>4</sub>H<sub>9</sub>O·CHO, m. 203° dec. <sup>73</sup>  
 PhCHO, m. 90°. <sup>73</sup>  
 PhCH:CHCHO, m. 175°. <sup>73</sup>  
 HOC<sub>6</sub>H<sub>4</sub>CHO, *o*, m. 300° dec.; *p*, m. 180°  
 dec. <sup>73</sup>  
*p*-MeOC<sub>6</sub>H<sub>4</sub>CHO, m. 115° dec. <sup>73</sup>  
 Vanillin, m. 125°. <sup>73</sup>  
 O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO, *o*, m. 125°; *m*, m. 160°; *p*,  
 m. 190° dec. <sup>73</sup>  
*p*-Me<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO, m. 175°. <sup>73</sup>  
 Diethyl, PhCHO, m. 74°. <sup>294</sup>  
*o*-MeOC<sub>6</sub>H<sub>4</sub>CHO, m. 85°. <sup>294</sup>  
 3-Cetyl, 2-Phenyl, PhCHO, m. 75°. <sup>242, 243</sup>  
 3-Phenyl, 2-Ethyl (or 2-Phenyl-3-ethyl), PhCHO, m. 97°. <sup>5</sup>  
 2-Allyl (or 2-Phenyl-3-allyl), PhCHO, m. 106°. <sup>5</sup>  
 Diphenyl, *o*-HOC<sub>6</sub>H<sub>4</sub>CHO, m. 230–5°. <sup>316</sup>  
*m*-C<sub>12</sub>H<sub>25</sub>OC<sub>6</sub>H<sub>4</sub>CHO, m. 124°. <sup>242</sup>  
 3-(2,4-Di-*t*-Am-phenoxyacetoxy)C<sub>6</sub>H<sub>3</sub>CHO, m. 133–  
 5°. <sup>242</sup>

- 3                      2
- m*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO, m. 225–7°. <sup>242</sup>
- m*-H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO, m. 280–2°. <sup>242</sup>
- 2,4-(HOOCCH<sub>2</sub>O)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>CHO, m. 157–9°. <sup>242</sup>
- o*-sulfoC<sub>6</sub>H<sub>4</sub>CHO, Na salt, m. >300°. <sup>242</sup>
- 3-(3-sulfobenzamido)C<sub>6</sub>H<sub>4</sub>CHO, dec. >175°. <sup>242</sup>
- Ditolyl, 2-C<sub>4</sub>H<sub>3</sub>O·CHO, *o*, m. 102°. <sup>28</sup>
- PhCHO, *o*, m. 182.5°, <sup>264</sup> 180°, <sup>356</sup> 95°; <sup>26, 28</sup> *m*, m. 176°; <sup>25</sup> *p*, m. 194°. <sup>24</sup>
- PhCH:CHCHO, *o*, m. 81°; <sup>28</sup> *m*, m. 184°; <sup>25</sup> *p*, m. 205°. <sup>24</sup>
- o*-HOC<sub>6</sub>H<sub>4</sub>CHO, *m*, m. 235°; <sup>25</sup> *p*, m. 257°. <sup>24</sup>
- m*-HOC<sub>6</sub>H<sub>4</sub>CHO, *m*, m. 169°; <sup>25</sup> *p*, m. 196°. <sup>24</sup>
- p*-MeOC<sub>6</sub>H<sub>4</sub>CHO, *o*, m. 131°; <sup>26</sup> *m*, m. 182°; <sup>25</sup> *p*, m. 210°. <sup>24</sup>
- 3,4-(MeO)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>CHO, *p*, m. 199°. <sup>24</sup>
- 3,4-CH<sub>2</sub>O<sub>2</sub>C<sub>6</sub>H<sub>3</sub>CHO, *m*, m. 252°; <sup>25</sup> *p*, m. 217°. <sup>24</sup>
- Vanillin, *m*, m. 220°. <sup>25</sup>
- p*-ClC<sub>6</sub>H<sub>4</sub>CHO, *o*, m. 147°. <sup>26</sup>
- o*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO, *m*, m. 164°; <sup>25</sup> *p*, m. 143°. <sup>24</sup>
- m*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO, *o*, m. 112°; <sup>28</sup> *m*, m. 208°; <sup>25</sup> *p*, m. 197°. <sup>24</sup>
- p*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO, *p*, m. 233°. <sup>24</sup>
- p*-Me<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO, *o*, m. 65°, <sup>26</sup> 132°. <sup>28</sup>
- Me<sub>2</sub>CO, m. 141°. <sup>26</sup>
- MeCOC<sub>6</sub>H<sub>4</sub>Cl-*p*, m. 147°. <sup>26</sup>
- Di-*o*-MeOC<sub>6</sub>H<sub>4</sub>, PhCHO, m. 160°. <sup>226</sup>
- Di-*o*-EtOC<sub>6</sub>H<sub>4</sub>, PhCHO, m. 144°. <sup>226</sup>
- Di-*p*-PhOC<sub>6</sub>H<sub>4</sub>, PhCHO, m. 151°. <sup>231</sup>
- (ClC<sub>6</sub>H<sub>4</sub>-)<sub>2</sub>, PhCHO, *o*, m. 170°; *m*, m. 172°; *p*, m. 212°. <sup>226</sup>
- Di-naphthyl, 2C<sub>4</sub>H<sub>3</sub>O·CHO, *α*, m. 180°; *β*, m. 238°. <sup>23</sup>
- PhCHO, *α*, m. 155°; *β*, m. 107°. <sup>23</sup>
- PhCH:CHCHO, *β*, m. 95°. <sup>23</sup>
- o*-HOC<sub>6</sub>H<sub>4</sub>CHO, *α*, m. 188°; *β*, m. 86°. <sup>23</sup>
- m*-HOC<sub>6</sub>H<sub>4</sub>CHO, *α*, m. 120°; *β*, m. 104°. <sup>23</sup>
- Anisaldehyde, *α*, m. 185°; *β*, m. 76°. <sup>23</sup>
- Vanillin, *α*, m. 186°; *β*, m. 210°. <sup>23</sup>
- m*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO, *α*, m. 195°; *β*, m. 77°. <sup>23</sup>
- p*-Me<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO, *α*, m. 158°; *β*, m. 105°. <sup>23</sup>
- 2-Benzoyl-3-*o*-PhOC<sub>6</sub>H<sub>4</sub>, *p*-Me<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO, m. 233°. <sup>231</sup>

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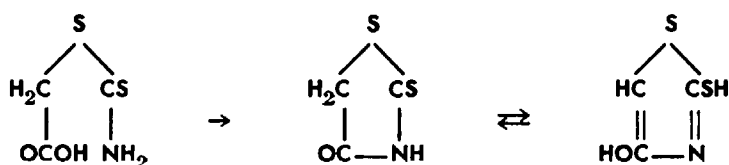
## CHAPTER 6

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# Rhodanine or Rhodanic Acid

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The reaction product of sodium chloracetate with ammonium dithiocarbamate is condensed by acids:



The product is 2-thio-4-ketothiazolidine or its tautomer, 2-mercapto-4-hydroxythiazole. Its two sets of reactions are appropriate to the two structures. It melts at 170° and is known as rhodanine, rhodanic acid, or rhodaninic acid.<sup>88, 89, 122, 134</sup>

### Preparation

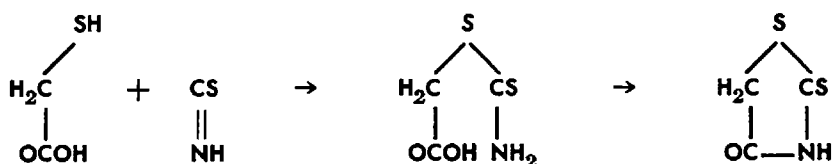
Sodium chloracetate is added to an aqueous solution of freshly prepared ammonium dithiocarbamate; strong acidification with hydrochloric acid precipitates the rhodanine.<sup>101, 151</sup> The condensation goes just as well with the ester,  $\text{H}_2\text{N}\cdot\text{CS}\cdot\text{S}\cdot\text{CH}_2\text{COOEt}$ ,<sup>43,5, 133</sup> as with the free acid,  $\text{H}_2\text{N}\cdot\text{CS}\cdot\text{S}\cdot\text{CH}_2\text{COOH}$ . By starting with the halo derivatives of higher acids, such as  $\alpha$ -bromopropionic acid, an alkyl group can be put into the 5-position.<sup>5, 52, 191</sup> Below 40° chloromalonate ester and ammonium dithiocarbamate give 5-carbethoxy rhodanine, but above 70°, the product is the unsubstituted rhodanine.<sup>40</sup>



Carboxymethyl trithiocarbamate with ammonia gives rhodanine; <sup>41</sup> with hydrazine the 3-amino derivative is obtained; <sup>26, 168, 201</sup> with a carboxylic acid hydrazide the product is a 3,5-derivative.<sup>164</sup> When hydrazine is treated with carbon disulfide and then with ethyl chloracetate the 3-amino-rhodanine is formed.<sup>5, 158, 186</sup> Glycylglycine put through the same series of reactions gives a rhodanine having the  $-\text{CH}_2\text{CONHCH}_2\text{COOH}$  group in the 3-position.<sup>5</sup> Analogous products are obtained by starting with other amino-acids.<sup>5, 203</sup> An alkyl on the nitrogen in the dithiocarbamic acid,  $\text{RNH}\cdot\text{CS}\cdot\text{SH}$ , is found in the 3-position in the 2-thio-3-alkylthiazolidine and blocks its isomerization to a thiazole.<sup>5, 6, 22, 103, 111</sup> Heating methyl thiocynoacetate and phenyl isothiocyanate with lead acetate until evolution of carbon dioxide ceases gives 3-phenylrhodanine.<sup>202</sup>

Optically active rhodanines have two dissimilar groups in the 5-position.<sup>102</sup>

Rhodanine can be prepared from other starting materials. It is formed when chloracetic acid is heated with an excess of ammonium thiocyanate; <sup>136, 137</sup> and when hydrogen chloride is passed into an alcoholic solution of thioglycolic acid and potassium thiocyanate:



The postulated intermediate,  $\text{H}_2\text{N}\cdot\text{CS}\cdot\text{SCH}_2\text{COOH}$ , is the starting material in the other method.<sup>65</sup> 5- and 5,3-substituted rhodanines may be prepared from chloracetic acid, ammonium thiocyanate, and an appropriate oxo compound.<sup>179</sup> A thiocynoacetic ester takes up hydrogen sulfide: <sup>134</sup>



An alcoholic solution of the ester is saturated with hydrogen sulfide and hydrochloric acid is added. Evaporation of this solution leaves rhodanine. Thiocynoacetic acid, or its ester, passes into rhodanine when treated with thioacetic acid. The addition of thioacetic acid and the elimination of acetic acid is equivalent to adding hydrogen sulfide and subtracting water.<sup>133, 191</sup> Evapo-

rating an ether solution of thiocyanoacetic acid and thiocyanic acid gives rhodanine.<sup>109,5</sup>

### Reactions

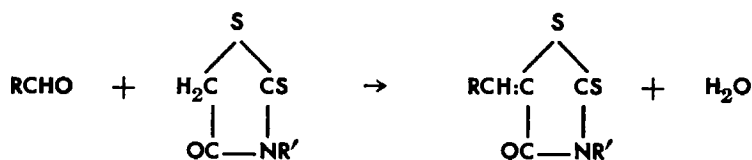
Rhodanic acid is hydrolyzed by alkali to thioglycolic anhydride and a thiocyanate salt,<sup>68,5, 137</sup> which may serve to identify it.<sup>189</sup> This has been suggested as a means for the manufacture of thioglycolic acid.<sup>83</sup> Hydrolysis of 5-isopropylidene rhodanine gives dimethyl  $\alpha$ -thiopyruvic acid;<sup>129</sup> 5-(arylmethylene) derivatives form  $\text{ArCH:C(SH)COOH}$ .<sup>29, 78, 114</sup> From 3-benzylidene aminorhodanine the product is  $\text{PhCH:NNHCS}_2\text{CH}_2\text{COOH}$ .<sup>186</sup> A methyl or isonitroso group in the 5-position reduces the rate of hydrolysis.<sup>181</sup> The ring stability of rhodanine and 5-methyl rhodanine depends on the substituent of position 2.<sup>184</sup> Aromatic amino acids can be prepared by the hydrolysis of the appropriately substituted rhodanines.<sup>68</sup> Hydrolysis of rhodanine with hydrochloric acid yields thioglycolic acid and 2,4-dioxothiazolidine.<sup>109,5</sup>

Rhodanine red,  $\text{C}_{13}\text{H}_6\text{O}_5\text{N}_4\text{S}_{10}$ ,<sup>109,5</sup> is formed by oxidation of rhodanine with feric chloride.<sup>72, 136</sup> *Bis*[5-(3-ethylrhodanine)] is formed by heating formamide with 3-ethylrhodanine at  $130^\circ$ .<sup>93</sup> Treating rhodanine with  $\text{CS}_2$  in the absence of water and further treatment with an alkylating agent puts  $\text{:C(SR)}_2$  in the 5-position.<sup>48</sup> Treatment of a rhodanine with an orthoformic ester puts an alkoxy group in the 5-position.<sup>36, 43, 111, 123, 124</sup> Auramine groups can be introduced into the 5-position.<sup>142</sup> Substitution in the 3-position may take place with 1,2-dimethyl-5,6-methylenedioxyindole.<sup>66</sup> 5-Phenylazo rhodanine is obtained by diazo coupling.<sup>77</sup>

Phenylhydrazine,<sup>130</sup> hydroxylamine,<sup>183</sup> and semicarbazide hydrochloride<sup>185</sup> react with the  $\text{:CS}$  group rather than with the carbonyl. A Grignard reagent attacks the double bond of a 5-Ar-alkylidene rhodanine.<sup>135</sup> Bromine substitutes in the 5-position of 3-*p*-tolyl-rhodanine.<sup>149</sup> 2-Rhodanine- $\beta$ -propionic acid is obtained by treatment of rhodanine with  $\beta$ -propiolactone.<sup>76</sup> Refluxing rhodanine and chloroacetic acid in solution gives the 2,4-dione.<sup>37</sup>

A characteristic reaction of rhodanine and of its N-substitution products, provided the 5-position is open, is the condensation

with aldehydes: 5, 6, 7.5, 31, 50, 69, 73, 85, 86, 96, 101, 103, 109, 136, 137, 146, 148, 160, 199



A number of these condensation products may be found in the tables of properties. Alloxan, oxindole, and isatin condense in the same way as aldehydes.<sup>27, 74, 100</sup> 3-Alkyl- or aryl-5-substituted methylene rhodanines are formed by the condensation of 3-substituted rhodanines and an orthoester.<sup>54, 197</sup>

### Use in Analysis

Rhodanine is of service in analytical chemistry.<sup>55, 99, 121</sup> It is specially recommended for silver in the presence of other metals.<sup>105, 131, 153</sup> The silver salt may be dried and weighed.<sup>59</sup> Certain of its derivatives are even more useful.<sup>46, 55, 107, 115, 116, 117, 180</sup> It can be used to identify aldehydes.<sup>143</sup>

*p*-Dimethylaminobenzilidene rhodanine has come into prominence as an analytical reagent.<sup>7</sup> It gives a red-violet coloration as precipitate with silver ions. Its preparation has been described.<sup>55, 56</sup> It is specific for silver,<sup>32, 53, 58, 70, 79, 98, 154, 158.5, 165, 175, 190</sup> particularly in the presence of potassium cyanide.<sup>84</sup> It can be used also for gold,<sup>57, 80, 84, 98</sup> mercury,<sup>13, 55, 113, 163</sup> and copper.<sup>67, 113</sup> It serves as an indicator in the volumetric determination of cyanides,<sup>139, 155</sup> and also in acid-base titrations in an acetic acid system.<sup>87</sup> The analogous diethyl compound has been recommended for the colorimetric estimation of gold.<sup>156</sup>

### Other Uses

Rhodanine derivatives have been claimed as photographic dyes.<sup>24, 26, 42, 44, 61, 111, 112, 119, 141, 187, 188</sup> Others have been considered as insecticides,<sup>9, 15, 33</sup> fungicides,<sup>4, 9, 16, 18, 75, 92, 106, 150</sup> bactericides,<sup>4, 91, 159, 171, 192</sup> and nematocides.<sup>12, 90, 172, 176</sup>

Rhodanine<sup>17, 94</sup> and several of its derivatives have been found to be effective against mildew in cotton duck, and are recommended as antioxidants,<sup>34</sup> stabilizers,<sup>63</sup> vulcanization accelerators,<sup>39, 49</sup> and as additives for extreme-pressure lubricants.<sup>110</sup>

Rhodanine slightly accelerates the auto-oxidation of methyl oleate;<sup>167</sup> and it is beneficial in both silver<sup>140</sup> and copper<sup>144</sup> plating baths.

### Physiological

The pharmacological and toxicological properties of rhodanine and some of its derivatives have been investigated.<sup>173, 200</sup> Some of them have been tested as anthelmintics.<sup>126, 127</sup> Rhodanine has anticonvulsant<sup>82</sup> and antithyroid<sup>28, 47, 81, 132</sup> activity, and inhibits the metamorphosis of tadpoles,<sup>132</sup> but it has no effect on blood sugar or on the histology of the pancreas in rabbits.<sup>132</sup> Certain derivatives of rhodanine have been tested for antiviral,<sup>38</sup> antitubercular,<sup>170</sup> and tyrosinase<sup>177</sup> activity. Its benzylidene derivative was found to be lethal to liver fluke.<sup>128</sup> Rhodanine-3-acetic acid activates the evolution of carbon dioxide from a yeast glucose fermentation.<sup>64</sup>

### Physical Properties

Rhodanine crystals grown from a solution in hot ethanol were hexagonal plates with well-formed {010} and {101} faces.<sup>133</sup> The infrared,<sup>195</sup> ultraviolet,<sup>125</sup> and X-ray spectra<sup>120</sup> of rhodanine have been studied, and so have the ultraviolet spectra of some of its 5-(arylmethylene) derivatives.<sup>29</sup> The ionization constants of *p*-dimethylaminobenzylidenerhodanine and the solubility product of silver *p*-diethylaminobenzylidenerhodanate have been determined.<sup>157</sup> At 25°C one liter of a saturated aqueous solution of rhodanine contains 2.25 gms.<sup>88</sup>

Rhodanine, m. 169°,<sup>158</sup> 169° dec.,<sup>88</sup> 168.5°,<sup>151</sup> 167°,<sup>133</sup> 168–70°;  
electrolytic dissociation constant  $K_{25^\circ} = 3 \times 10^{-6}$ .<sup>136</sup>

### 3-SUBSTITUTED RHODANINES

Methyl, m. 72°. <sup>5.5</sup>

Ethyl, oil. <sup>5.5</sup>

*i*-Propyl, m. 57–9°. <sup>148.5</sup>

*i*-Butyl, oil,  $b_{11-12}$  160°. <sup>135.5</sup>

*i*-Amyl, oil,  $b_{11}$  200°. <sup>162</sup>

*i*-Hexyl, oil,  $b_{11}$  199–200°. <sup>103</sup>

3,5,5-Trimethylhexyl, m. 41–3°. <sup>124</sup>

- Allyl, m.  $55^{\circ}$ ,<sup>111</sup>  $42^{\circ}$ ,<sup>202</sup> 46–48°; b<sub>12</sub> about  $186^{\circ}$ .<sup>27</sup>  
c-Hexyl, m.  $140^{\circ}$ .<sup>111</sup>  
2-Methoxyethyl, m.  $49^{\circ}$ .<sup>148.5</sup>  
Carboxymethyl, m.  $148^{\circ}$ ,<sup>112.5</sup>  $145^{\circ}$ .<sup>5</sup>  
Carbethoxymethyl, m.  $102^{\circ}$ ,<sup>111</sup>  $58^{\circ}$ ,<sup>112.5</sup>  $58.5^{\circ}$ .<sup>88</sup>  
Carboxyethyl, m.  $133^{\circ}$ .<sup>78</sup>  
 $\alpha$ -Carboxyethyl, m.  $147^{\circ}$ .<sup>5</sup>  
2-Benzothiazolyl methyl, m.  $122^{\circ}$ .<sup>201</sup>  
Phenyl, m.  $195^{\circ}$ ,<sup>89</sup>  $193^{\circ}$ ,<sup>5.5, 88, 202</sup>  $188^{\circ}$ ,<sup>16.5</sup> 164–6°; <sup>111</sup> Br<sub>2</sub> addition compound, m.  $143^{\circ}$ .<sup>5</sup>  
Chlorophenyl, *o*, m.  $117.5^{\circ}$ ; *m*, m.  $161^{\circ}$ ; <sup>22</sup> *p*, m.  $128^{\circ}$ .<sup>148.5</sup>  
2,5-Dichlorophenyl, m. 152–4°.<sup>22</sup>  
Bromophenyl, *o*, m.  $137^{\circ}$ ; *m*, m.  $179^{\circ}$ ; <sup>22</sup> *p*, m.  $165^{\circ}$ .<sup>88</sup>  
*p*-Iodophenyl, m.  $163^{\circ}$  dec.<sup>22</sup>  
*p*-Fluorophenyl, m.  $167.5^{\circ}$ .<sup>22</sup>  
2-Hydroxy-5-nitrophenyl, m.  $226^{\circ}$  dec.<sup>22</sup>  
Methoxyphenyl, *o*, m.  $143^{\circ}$ ; *p*, m.  $156^{\circ}$ ,<sup>88</sup>  $153^{\circ}$ .<sup>6</sup>  
*p*-Ethoxyphenyl, m. 180–88°.<sup>189.5</sup>  
*p*-Nitrophenyl, m.  $145^{\circ}$ .<sup>22</sup>  
*p*-Acetoxyphenyl, m.  $148^{\circ}$ .<sup>22</sup>  
Carboxyphenyl, *m*, m.  $270^{\circ}$  dec.; <sup>22</sup> *p*, m. 268–70°.<sup>26</sup>  
*p*-H<sub>2</sub>NSO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>, m. >240° dec.<sup>22</sup>  
Tolyl, *o*, m.  $114^{\circ}$ ,<sup>88</sup>  $101^{\circ}$ ; *m*, m.  $148^{\circ}$ ; *p*, m.  $160^{\circ}$ .<sup>5.5</sup>  
Benzyl, m.  $87^{\circ}$ ,<sup>111</sup>  $83^{\circ}$ .<sup>5</sup>  
*o*-Chlorobenzyl, m.  $88^{\circ}$ .<sup>22</sup>  
Dichlorobenzyl, 2,4, m.  $104^{\circ}$ ; 3,4, m.  $106^{\circ}$ .<sup>22</sup>  
*p*-Bromobenzyl, m.  $97^{\circ}$ .<sup>22</sup>  
*p*-Iodobenzyl, m.  $123^{\circ}$ .<sup>22</sup>  
*p*-Fluorobenzyl, m.  $65^{\circ}$ .<sup>22</sup>  
*p*-Methoxybenzyl, m.  $99.5^{\circ}$ .<sup>22</sup>  
*o*-Methylmercaptobenzyl, m. 119–21°.<sup>22</sup>  
*m*-( $\alpha,\alpha,\alpha$ -Trifluorotolyl), m.  $177^{\circ}$ .<sup>22</sup>  
3-Chloro-2-methylphenyl, m.  $122^{\circ}$ .<sup>22</sup>  
4-Chloro-2-methylphenyl, m.  $146^{\circ}$ .<sup>22</sup>  
2-Bromo-4-methylphenyl, m.  $119^{\circ}$ .<sup>22</sup>  
4-Bromo-2-methylphenyl, m.  $136^{\circ}$ .<sup>22</sup>  
Methylbenzyl, *o*, m.  $123^{\circ}$ ; *m*, m.  $64^{\circ}$ ; *p*, m.  $75^{\circ}$ .<sup>22</sup>  
 $\alpha$ -Phenethyl, *D*, m.  $109^{\circ}$ ; [ $\alpha$ ]<sub>D</sub>, +301.2°; *L*, m.  $109^{\circ}$ ; [ $\alpha$ ]<sub>D</sub>, –304.8°; <sup>102</sup> *DL*, m.  $112^{\circ}$ ,<sup>88</sup>  $111^{\circ}$ .<sup>102</sup>  
Dimethylphenyl, 2,4-, oil; <sup>5.5</sup> 2,5-, m.  $136^{\circ}$ ; 2,6-, –163°.<sup>22</sup>

*p*-Chlorophenylethyl, m. 109°. <sup>22</sup>  
 Naphthyl,  $\alpha$ , m. 168°;  $\beta$ , m. 180–90°. <sup>189.5</sup>  
 2-Furfuryl, m. 74°. <sup>22</sup>  
 Pyridyl, 2-, m. 77°; 3-, m. 157°; 4-, m. 163°. <sup>111</sup>  
 6-Methyl-2-pyridyl, m. 146–8°. <sup>111</sup>  
 3-Quinolyl, m. 228°. <sup>111</sup>  
 2-Thenyl, m. 95°. <sup>22</sup>  
 5-Chloro-2-thenyl, m. 93.5°. <sup>22</sup>  
 2-Thiazolyl, m. 130°. <sup>111</sup>  
 2-Methyl-5-benzothiazolyl, m. 236°. <sup>201</sup>  
 2-Methyl-6-Benzothiazolyl, m. 202°. <sup>201</sup>  
 Amino, m. 92.5°, <sup>188</sup> 92°, <sup>5, 158</sup> 94–6°. <sup>188</sup>  
 Anilino, m. 125°, <sup>5</sup> 134°. <sup>88</sup>  
 Benzamido, m. 182–3°. <sup>164</sup>  
*p*-Nitrobenzamido, m. 209°. <sup>164</sup>  
 Isonicotinamido, m. 196–8°. <sup>164</sup>  
 Benzylideneamino, m. 138°. <sup>158</sup>  
 Vanillylideneamino, m. 186–9°. <sup>158</sup>  
 Cinnamylideneamino, m. 146°. <sup>158</sup>

## 5-SUBSTITUTED RHODANINES

Methyl, m. 126°, <sup>158</sup> 124°, <sup>191</sup> 123.5°, <sup>88</sup> 123°. <sup>10.5, 43.5</sup>  
 Ethyl, m. 105°. <sup>5</sup>  
 Carboxymethyl, m. 161°. <sup>158</sup>  
 2-Benzoylvinyl, m. 255° dec. <sup>198</sup>  
 Phenyl, m. 183°, <sup>158</sup> 178–80°. <sup>174</sup>  
 Phenylazo, m. 234–6° dec. <sup>77</sup>  
 Anisylazo, *o*, m. 216° dec.; *p*, m. 188–90° dec. <sup>77</sup>  
*p*-Ethoxyphenylazo, m. 207–9°. <sup>77</sup>  
*o*-Carboxyphenylazo, m. 249° dec. <sup>77</sup>  
*p*-Dimethylaminophenylazo, m. 125° dec. <sup>77</sup>  
*p*-Diethylaminophenylazo, m. 131–3° dec. <sup>77</sup>  
*p*-Tolylazo, m. 226–8°. <sup>77</sup>

## FROM ALDEHYDES (5-SUBSTITUTED)

Methoxymethylene, m. 198°. <sup>124</sup>  
 Ethoxymethylene, m. 158°. <sup>124</sup>  
 2-Carboxy-4,5-methylene, m. 260° dec. <sup>23</sup>  
 Diethylaminomethylene, m. 213°. <sup>123</sup>  
 Dibutylaminomethylene, m. 141°. <sup>123</sup>

- 1,1,3,3-Tetramethylbutylaminomethylene, m. 176°. <sup>123</sup>  
 Allylaminomethylene, m. 157.5°. <sup>123</sup>  
 Cyclohexylaminomethylene, m. 185°. <sup>123</sup>  
 Dicyclohexylaminomethylene, m. 236.5°. <sup>123</sup>  
 Anilinomethylene, m. 226° dec. <sup>123</sup>  
 Acetanilidomethylene, m. 249° dec. <sup>123</sup>  
 6-Safrolylmethylene, m. 207.5°. <sup>166</sup>  
 Myristicinylmethylene, m. 255°. <sup>152</sup>  
 $\beta$ -Naphthylmethylene, m. 281°. <sup>1</sup>  
 Anthrylmethylene, m. 298°. <sup>1</sup>  
 Phenanthrylmethylene, m. 285°. <sup>1</sup>  
 Morpholinomethylene, m. 266° dec. <sup>123</sup>  
 Piperidinomethylene, m. 253° dec. <sup>123</sup>  
 Quinolinomethylene, 2, m. 270°; 4, m. 318–20°. <sup>2</sup>  
 Indolylmethylene, m. 287°. <sup>138</sup>  
 10-Methyl-3-phenothiazinylmethylene, m. 248° dec. <sup>126</sup>  
 3-Thianaphthenylmethylene, m. 241°. <sup>35</sup>  
*bis*-(2-Hydroxyethyl)aminomethylene, m. 182–5° dec. <sup>123</sup>  
*bis*-(*p*-Dimethylaminophenyl)methylene, m. 224°. <sup>142</sup>  
 Ethylidene, m. 148°. <sup>136</sup>  
 Isopropylidene, m. 199°, <sup>158</sup> 197°. <sup>129</sup>  
 2-Ethylbutylidene, m. 104–6°. <sup>20</sup>  
 2-Ethylhexylidene, m. 66–8°. <sup>20</sup>  
 Decylidene, m. 76.5°. <sup>20</sup>  
 Citrylidene, m. 147°. <sup>20</sup>  
 Furylacrylidene, m. 205°. <sup>147</sup>  
 2-Ethyl-3-propylallylidene, m. 110.5°. <sup>20</sup>  
 3-(2-Furylallylidene), m. 251° dec. <sup>20</sup>  
 Furylpentadienylidene, m. 200°. <sup>147</sup>  
 Benzylidene, m. 202.5°, <sup>118</sup> 200°. <sup>7.5, 68, 136</sup>  
 Dichlorobenzylidene, 2,4-, m. 232.5°; 3,4-, m. 231°. <sup>20</sup>  
 5-Chloro-2-hydroxybenzylidene, m. 223° dec. <sup>20</sup>  
 Fluorobenzylidene, *o*, m. 201–3°; *m*, m. 201°; *p*, m. 227°. <sup>20</sup>  
*p*-Acetamidobenzylidene, m. 290° dec., <sup>169</sup> 285–8°. <sup>198</sup>  
 Dimethylaminobenzylidene, *m*, m. 270° dec.; <sup>126</sup> *p*, m. 273°. <sup>118</sup>  
 Hydroxybenzylidene, *o*, m. 218–20° dec., <sup>126</sup> 219°, <sup>7.5</sup> 216°, <sup>118</sup>  
 200°; <sup>199</sup> *m*, m. 245°; <sup>7.5</sup> *p*, m. 275–7°, <sup>114</sup> 260° dec., <sup>7.5</sup> 258–60°  
 dec. <sup>126</sup>  
*o*-MeCOOC<sub>6</sub>H<sub>4</sub>CH:, m. 202°, <sup>5.5</sup> 168°. <sup>199</sup>

- 3,4-Dihydroxybenzylidene, m. 315–9°. <sup>11</sup>
- p*-Methoxybenzylidene, m. 250° dec., <sup>126</sup> 230–42°. <sup>7.5</sup>
- 2-Hydroxy-3-methoxybenzylidene, m. 240°. <sup>20</sup>
- Vanillylidene, m. 230°, <sup>97</sup> 227–30°, <sup>7.5</sup> 228–30°, <sup>95</sup> 228°, <sup>62</sup> 225°. <sup>118</sup>
- Isovanillylidene, m. 221°. <sup>78</sup>
- Piperonylidene, m. 255° dec. <sup>126</sup>
- 4-Hydroxy-3-ethoxybenzylidene, m. 216°. <sup>20</sup>
- Dimethoxybenzylidene, 2,3-, m. 209°; <sup>8</sup> 2,4-, m. 275°, <sup>50</sup> 267–70°; <sup>51</sup> 2,6-, m. 267°. <sup>108</sup>
- 3,4-Diethoxybenzylidene, m. 196°. <sup>20</sup>
- Carboxybenzylidene, *o*, m. 266°; <sup>45</sup> *p*, m. 308°. <sup>2</sup>
- 6-Carbomethoxy-2,4-dimethoxybenzylidene, m. 217.5°. <sup>104</sup>
- 2-Carbomethoxy-3,4-dimethoxybenzylidene, m. 191–3°. <sup>45</sup>
- 2-Carboxy-4,5-methylenedioxybenzylidene, m. 256°. <sup>23</sup>
- Nitrobenzylidene, *o*, m. 205°, <sup>2</sup> 189°; <sup>14</sup> *m*, m. 271°, <sup>96</sup> 265°, <sup>2</sup> 245–55°, <sup>7.5</sup> 225–8°; <sup>198</sup> *p*, m. 274°, <sup>2</sup> 250–52° dec. <sup>14</sup>
- Methylbenzylidene, *o*, m. 196°; *m*, m. 182–5°; *p*, m. 220°. <sup>20</sup>
- $\alpha$ -(2-Carboxyethyl)benzylidene, m. 176–8°. <sup>3</sup>
- Cinnamylidene, m. 221°, <sup>7.5</sup> 220–5° dec., <sup>126</sup> 219°, <sup>60, 147</sup> 208–11°. <sup>190</sup>
- o*-Nitrocinnamylidene, m. 250°, <sup>20</sup> 248–50°. <sup>126</sup>
- $\alpha$ -Ethylcinnamylidene, m. 166°. <sup>60</sup>
- $\alpha$ -Amylcinnamylidene, m. 145–8°. <sup>20</sup>
- $\alpha$ -Phenylcinnamylidene, m. 228–30°. <sup>60</sup>
- o*-Methylcinnamylidene, m. 204–6°. <sup>60</sup>
- Ph(CH:CH)<sub>2</sub>CH:, m. 219°. <sup>193</sup>
- Ph(CH:CH)<sub>3</sub>CH:, m. 239°. <sup>193</sup>
- Cuminylidene, m. 204°, <sup>135.5</sup> 154–7°. <sup>7.5</sup>
- 3-Nitrocuminylidene, m. 180°. <sup>145.5</sup>
- 4,2,5-HO(Me)(Me<sub>2</sub>CH)C<sub>6</sub>H<sub>2</sub>CH:, m. 221°. <sup>7.5</sup>
- 4,2,5,3-HO(Me)(Me<sub>2</sub>CH)(formyl)C<sub>6</sub>HCH:, m. 240°. <sup>7.5</sup>
- Furfurylidene, m. 234°, <sup>148.5</sup> 229–31° dec., <sup>126</sup> 224–6°. <sup>118</sup>
- 5-Chlorofurfurylidene, m. 220° dec. <sup>20</sup>
- 5-Nitrofurfurylidene, m. 201.5°; <sup>20</sup> Ac., m. 195°. <sup>69</sup>
- 5-Methyl-4-carbethoxyfurfurylidene, m. 242°. <sup>69</sup>
- Pyridylidene, 2-, m. 253–8°, <sup>138</sup> 247–50° dec., <sup>118</sup> 243–5°; 3-, m. 318–20°, <sup>2</sup> 300° dec., <sup>118</sup> 285–8°; <sup>138</sup> 4-, m. 320–2°, <sup>2</sup> 295–305°, <sup>138</sup> 279° dec. <sup>118</sup>
- 2-Pyrrole CH:, m. 286°, <sup>86</sup> 159°. <sup>85</sup>
- Thenylidene, m. 232°, <sup>35</sup> 213°. <sup>30</sup>



- 3,4-Dimethylthénylidene, m. 264°. <sup>35</sup>  
 3,4,5-Trimethylthénylidene, m. 276°. <sup>35</sup>  
 2-Methylsénylidene, m. 236°. <sup>196</sup>

FROM KETONES (5-SUBSTITUTED)

*Ketone*

- Acetone, m. 197°. <sup>21</sup>  
 MeCOH<sub>2</sub>CSMe, m. 110.5°. <sup>16</sup>  
 MeCOH<sub>2</sub>CSEt, m. 128°. <sup>16</sup>  
 MeCOH<sub>2</sub>CSPr, m. 91.5°. <sup>16</sup>  
 MeCOH<sub>2</sub>CSPr-*i*, m. 112.5°. <sup>16</sup>  
 MeCOH<sub>2</sub>CSBu, m. 107.5°. <sup>16</sup>  
 MeCOH<sub>2</sub>CSBu-*i*, m. 112.5°. <sup>16</sup>  
 MeCOH<sub>2</sub>CSBu-*t*, m. 128°. <sup>16</sup>  
 MeCOH<sub>2</sub>CSAm, m. 91.5°. <sup>16</sup>  
 MeCOH<sub>2</sub>CSHex, m. 92°. <sup>16</sup>  
 MeCOEt, m. 120°. <sup>16</sup>  
 MeCOPr, m. 121°. <sup>21</sup>  
 MeCOPr-*i*, m. 192.5°. <sup>21</sup>  
 MeCOBu, m. 119°. <sup>21</sup>  
 MeCOBu-*i*, m. 99°. <sup>21</sup>  
 MeCONon, m. 107°. <sup>21</sup>  
 MeCOAm, m. 122°. <sup>21</sup>  
 MeCOHex, m. 108°. <sup>21</sup>  
 MeCOPh, m. 166°. <sup>21</sup>  
 MeCOC<sub>6</sub>H<sub>4</sub>Cl-*p*, m. 204°. <sup>21</sup>  
 MeCOC<sub>6</sub>H<sub>4</sub>Br-*p*, m. 216° dec. <sup>21</sup>  
 MeCOCH<sub>2</sub>Ph, m. 155°. <sup>21</sup>  
 Methyl styryl ketone, m. 192–4°. <sup>21</sup>  
 MeCOC<sub>6</sub>H<sub>4</sub>Me-*p*, m. 176°. <sup>21</sup>  
 MeCOCH<sub>2</sub>OC<sub>10</sub>H<sub>7</sub>-β, m. 188–90°. <sup>21</sup>  
 Methyl furyl ketone, m. 244.5°. <sup>21</sup>  
 MeCO(5-Me-2-furyl), m. 248° dec. <sup>21</sup>  
 Methyl furylvinyl ketone, m. 210–4°. <sup>21</sup>  
 Methyl 2-thienyl ketone, m. 218–21.5° dec. <sup>21</sup>  
 MeCO(5-Cl-2-thienyl), m. 205°. <sup>21</sup>  
 MeCO(5-Br-2-thienyl), m. 215° dec. <sup>21</sup>  
 MeCO(5-*t*-Bu-2-thienyl), m. 193°. <sup>21</sup>  
 Diethyl ketone, m. 123°. <sup>21</sup>  
 Ethyl phenyl ketone, m. 147°. <sup>21</sup>

*Ketone*Ethyl furyl ketone, m.  $181^{\circ}$ .<sup>21</sup>Propyl furyl ketone, m.  $160.5^{\circ}$ .<sup>21</sup>Cyclopentanone, m.  $196.5^{\circ}$ ,<sup>178</sup>  $196^{\circ}$ .<sup>21</sup>Cyclohexanone, m.  $173^{\circ}$ ,<sup>21</sup>  $170^{\circ}$ .<sup>178</sup>Methylcyclohexanone, 2-, m.  $141-3^{\circ}$ ; 3-, m.  $146-8^{\circ}$ ; 4-, m.  $150^{\circ}$ .<sup>21</sup>4-(*t*-Amylcyclohexanone), m.  $183^{\circ}$ .<sup>21</sup>

## 3,5-DISUBSTITUTED RHODANINES

R-3      R-5

Methyl, Ethyl, m.  $62^{\circ}$ .<sup>148.5</sup>Ethyl,  $\text{HOOCCH}_2$ , m.  $119^{\circ}$ .<sup>102</sup> $\text{H}_2\text{NCOCH}_2$ , m.  $108^{\circ}$ .<sup>102</sup>*i*-Propyl, Phenyl, m.  $122.5-4^{\circ}$ .<sup>174</sup>Allyl, Methyl, m.  $64^{\circ}$ .<sup>111</sup>Ethyl, m.  $63^{\circ}$ .<sup>111</sup>Hydroxyimino, m.  $148^{\circ}$ .<sup>111</sup>*c*-Hexyl, Methyl, m.  $126^{\circ}$ .<sup>111</sup>Carbethoxymethyl, Methyl, m.  $105^{\circ}$ .<sup>111</sup>Ethyl, m.  $98^{\circ}$ .<sup>111</sup>Phenyl, Methyl, m.  $119^{\circ}$ ,<sup>5.5</sup>  $117^{\circ}$ .<sup>88</sup>Ethyl, m.  $180-2^{\circ}$ ,<sup>111</sup>  $83^{\circ}$ .<sup>88</sup>*i*-Propyl, m.  $84^{\circ}$ .<sup>111, 182</sup>Phenyl, m.  $230^{\circ}$ .<sup>174</sup>*p*-Chlorophenyl, Methyl, m.  $117^{\circ}$ .<sup>9</sup>Ethyl,  $89^{\circ}$ .<sup>9</sup>Benzyl, Methyl, m.  $109^{\circ}$ .<sup>111</sup>*p*-Tolyl, Bromo, m.  $>300^{\circ}$ .<sup>149</sup>Amino, Carboxymethyl, m.  $82^{\circ}$ .<sup>158</sup>Acetyl, m.  $141^{\circ}$  dec.<sup>158</sup>Phenyl, m.  $183^{\circ}$ .<sup>158</sup>*p*-Dimethylaminobenzyl, m.  $230-2^{\circ}$ .<sup>186</sup> $\alpha$ -Phenylaminobenzyl, m.  $191-3^{\circ}$ .<sup>186</sup>Benzylideneamino, Carboxymethyl, m.  $190^{\circ}$ .<sup>158</sup>Phenyl, m.  $157^{\circ}$ .<sup>158</sup>Veratrylideneamino, Methyl, m.  $140^{\circ}$ .<sup>158</sup>

## FROM ALDEHYDES AND KETONES (3,5-DISUBSTITUTED)

Methyl, Ethoxymethylene, m.  $133^{\circ}$ .<sup>124</sup>Aminomethylene, m.  $175^{\circ}$ .<sup>123</sup>

- Cyclopentylidene, m. 108.5°. <sup>20</sup>  
 Cyclohexylidene, m. 112°. <sup>20</sup>  
 4-Methylcyclohexylidene, m. 106°. <sup>20</sup>  
 2-Thenylidene, m. 170.5°. <sup>20</sup>  
 Benzylidene, m. 170°. <sup>20</sup> 169°. <sup>5.5</sup>  
*p*-Chlorobenzylidene, m. 198–200°. <sup>20</sup> 196–8°. <sup>148.5</sup>  
 Nitrobenzylidene, *m*, m. 233°; *p*, m. 205°. <sup>5.5</sup>  
 Anisylidene, m. 181°. <sup>5.5</sup>  
 Vanillylidene, m. 199°. <sup>5.5</sup>  
 Methylbenzylidene, *m*, m. 164–7°; *p*, m. 170°. <sup>20</sup>  
*p*-Isopropylbenzylidene, m. 138°. <sup>20</sup>  
 Cinnamylidene, m. 226°. <sup>5.5</sup>  
 Ethyl, Ethoxymethylene, m. 92.5°. <sup>123</sup>  
 Acetylanilinomethylene, m. 133° dec. <sup>43</sup>  
*bis*-(*p*-Dimethylaminophenyl)methylene, m. 227°. <sup>142</sup>  
 Naphthylmethylene,  $\alpha$ , m. 143°;  $\beta$ , m. 177°. <sup>1</sup>  
 9-Anthrylmethylene, m. 191°. <sup>1</sup>  
 9-Phenanthrylmethylene, m. 182°. <sup>1</sup>  
 2-(6-Formyl)pyridylmethylene, m. 248–50°. <sup>2</sup>  
 Ethylaminoethylidene, m. 113°. <sup>111</sup>  
 Cyclopentylidene, m. 101.5°. <sup>25</sup>  
 Benzylidene, m. 150.5°. <sup>88</sup> 149°. <sup>5.5</sup>  
 Nitrobenzylidene, *o*, m. 158°; *m*, m. 195°; <sup>2</sup> 188°; <sup>5.5</sup> *p*, m. 233°. <sup>2</sup>  
 Salicylidene, m. 190°. <sup>5.5</sup>  
 Anisylidene, m. 143°. <sup>5.5</sup>  
 Vanillylidene, m. 140°. <sup>5.5</sup>  
 Carboxybenzylidene, *o*, m. 185.7°; *p*, m. 290–1°. <sup>2</sup>  
 Cinnamylidene, m. 187°. <sup>5.5</sup>  
 4-Formylbenzylidene, m. 172°. <sup>2</sup>  
 1-Methyl-2-pyridylidene, m. 250–2°. <sup>119</sup>  
*n*-Butyl, Furfurylidene, m. 104°. <sup>148.5</sup>  
*p*-Chlorobenzylidene, m. 150°. <sup>148.5</sup>  
*i*-Butyl, Benzylidene, m. 117°. <sup>135.5</sup>  
 Hydroxybenzylidene, *o*, m. 184°; *p*, m. 153°. <sup>135.5</sup>  
*i*-Amyl, Benzylidene, m. 105°. <sup>162</sup>  
*p*-Nitrobenzylidene, m. 163°. <sup>162</sup>  
 Hydroxybenzylidene, *o*, m. 175°; *p*, m. 161°. <sup>162</sup>  
 Anisylidene, m. 116°. <sup>162</sup>

- i*-Hexyl, Benzylidene, m.  $87^{\circ}$ .<sup>103</sup>  
     Nitrobenzylidene, *m*, m.  $167^{\circ}$ ; *p*, m.  $131^{\circ}$ .<sup>103</sup>  
     Salicylidene, m.  $170-2^{\circ}$ .<sup>103</sup>  
     Anisylidene, m.  $85^{\circ}$ .<sup>103</sup>  
 3,5,5-Trimethylhexyl, Ethoxymethylene, m.  $39-41^{\circ}$ .<sup>124</sup>  
 Allyl, Aminomethylene, m.  $104-9^{\circ}$ .<sup>111</sup>  
     Carboxymethylaminomethylene, m.  $196^{\circ}$ .<sup>111</sup>  
     Ethylaminomethylene, m.  $132^{\circ}$ .<sup>111</sup>  
     Diethylaminomethylene, m.  $74^{\circ}$ .<sup>111</sup>  
     Octylaminomethylene, m.  $98-100^{\circ}$ .<sup>111</sup>  
     Cyclohexylaminomethylene, m.  $133-4^{\circ}$ .<sup>111</sup>  
     Anilinomethylene, m.  $162^{\circ}$ .<sup>54, 111</sup>  
     *bis*-(*p*-Dimethylaminophenyl)methylene, m.  $208^{\circ}$ .<sup>142</sup>  
     Morpholinomethylene, m.  $180^{\circ}$ .<sup>111</sup>  
     10-Methyl-3-phenothiazinylmethylene, m.  $162-4^{\circ}$ .<sup>126</sup>  
     Methoxyethylidene, m.  $84^{\circ}$ .<sup>111</sup>  
     Ethylaminoethylidene, m.  $65^{\circ}$ .<sup>111</sup>  
     Ethylaminopropylidene, m.  $49-51^{\circ}$ .<sup>111</sup>  
     Cyclohexylidene, m.  $64^{\circ}$ .<sup>20</sup>  
     *p*-Methylcyclohexylidene, m.  $49-51^{\circ}$ .<sup>20</sup>  
     2-Furfurylidene, m.  $103.5^{\circ}$ ,<sup>20</sup>  $102^{\circ}$ .<sup>202</sup>  
     2-Thenylidene, m.  $149^{\circ}$ .<sup>20</sup>  
     Benzylidene, m.  $144^{\circ}$ .<sup>5.5, 202</sup>  
     *p*-Chlorobenzylidene, m.  $138^{\circ}$ .<sup>20</sup>  
     *o*-Hydroxybenzylidene, m.  $180^{\circ}$ ,<sup>202</sup>  $179^{\circ}$ .<sup>5.5</sup>  
     Nitrobenzylidene, *o*, m.  $73^{\circ}$ ; <sup>5.5</sup> *m*, m.  $148^{\circ}$ ,<sup>202</sup>  $145^{\circ}$ ; *p*, m.  $153^{\circ}$ .<sup>5.5</sup>  
     Anisylidene, m.  $114^{\circ}$ .<sup>5.5</sup>  
     Vanillylidene, m.  $148^{\circ}$ .<sup>5.5</sup>  
     *p*-Methylbenzylidene, m.  $126^{\circ}$ .<sup>20</sup>  
     Cinnamylidene, m.  $176-8^{\circ}$ ,<sup>202</sup>  $166^{\circ}$ .<sup>5.5</sup>  
     *p*-Isopropylbenzylidene, m.  $49^{\circ}$ .<sup>20</sup>  
      $\alpha$ -Naphthylidene, m.  $111-3^{\circ}$ .<sup>202</sup>  
      $\beta$ -Hydroxynaphthylidene,  $\alpha$ , m.  $111-13^{\circ}$ ;  $\beta$ , m.  $196^{\circ}$ .<sup>202</sup>  
 Carbethoxymethyl, Benzylaminomethylene, m.  $110^{\circ}$ .<sup>111</sup>  
     Dibenzylaminomethylene, m.  $104^{\circ}$ .<sup>111</sup>  
     Methylanilinomethylene, m.  $146-9^{\circ}$ .<sup>111</sup>  
     Piperidinomethylene, m.  $147^{\circ}$ .<sup>111</sup>  
     Morpholinomethylene, m.  $173^{\circ}$ .<sup>111</sup>

- Methoxyethylidene, m. 114.5°. <sup>111</sup>  
 Ethylaminoethylidene, m. 121°. <sup>111</sup>  
 Dicyclohexylaminoethylidene, m. 159°. <sup>111</sup>  
 $\alpha$ -Phenylaminoethylidene, m. 173-5°. <sup>111</sup>  
 CH<sub>2</sub>CONHCH<sub>2</sub>COOH, Benzylidene, m. 190°. <sup>5</sup>  
 CHMeCOOH, Benzylidene, m. 191°. <sup>5</sup>  
 Phenyl, Ethoxymethylene, m. 153-5°. <sup>124</sup>  
   Naphthylmethylene,  $\alpha$ , m. 159°;  $\beta$ , m. 237°. <sup>1</sup>  
   Pyridylmethylene, 3-, m. 235-70°; 4-, m. 246°. <sup>2</sup>  
   9-Anthrylmethylene, m. 231°. <sup>1</sup>  
   9-Phenanthrylmethylene, m. 262-4°. <sup>1</sup>  
   2-Benzothiazolylaminomethylene, m. 258-60°. <sup>123</sup>  
   Anilinomethylene, m. 247°. <sup>123</sup>  
   2-Thienylmethylene, m. 160.5°. <sup>10</sup>  
   Propylidene, m. 104.5°. <sup>20</sup>  
   Isopropylidene, m. 137°. <sup>20</sup>  
   1-Methylhexylidene, m. 99°. <sup>20</sup>  
   Cyclohexylidene, m. 128°. <sup>20</sup>  
   4-Methylcyclohexylidene, m. 119°. <sup>20</sup>  
   Furfurylidene, m. 183°. <sup>202</sup>  
   Thenylidene, m. 198-200°. <sup>20</sup>  
   Benzylidene, m. 187-9°, <sup>202</sup> 186°. <sup>5.5</sup>  
   Hydroxybenzylidene, *o*, m. 180°, <sup>202</sup> 172°; <sup>5.5</sup> *p*, m. 285°. <sup>5</sup>  
   Anisylidene, m. 221°. <sup>5.5</sup>  
   Vanillylidene, m. 193°. <sup>5.5</sup>  
   Nitrobenzylidene, *o*, m. 246°, <sup>2</sup> 238°; <sup>5.5</sup> *m*, m. 246°, <sup>2</sup>  
     243.5°, <sup>202</sup> 240°; <sup>5.5</sup> *p*, m. 274°, <sup>2</sup> 264°, <sup>202</sup> dec. 240°. <sup>5.5</sup>  
   *p*-Acetamidobenzylidene, m. >284°. <sup>202</sup>  
   Carboxybenzylidene, *o*, m. 226°; *p*, m. 308°. <sup>2</sup>  
   *p*-Formylbenzylidene, m. 234°. <sup>2</sup>  
   *m*-Tolualdehyde, m. 200°. <sup>135.5</sup>  
   Cinnamylidene, m. 222°, <sup>202</sup> 217°. <sup>5.5</sup>  
    $\alpha$ -Naphthylidene, m. 145-7°. <sup>202</sup>  
   *p*-Dimethylaminophenylimino, m. 206°. <sup>114.5</sup>  
*o*-Hydroxyphenyl, Benzylidene, m. 211°. <sup>5.5</sup>  
*p*-Methoxyphenyl, Furfurylidene, m. 185°. <sup>148.5</sup>  
   Benzylidene, m. 190°. <sup>6</sup>  
   *p*-Hydroxybenzylidene, m. 258°. <sup>6</sup>  
   Vanillylidene, m. 210°. <sup>6</sup>  
   *p*-Chlorobenzylidene, m. 229-34°. <sup>148.5</sup>

- p*-Ethoxyphenyl, Benzylidene, m.  $212^{\circ}$ .<sup>189.5</sup>  
*p*-Chlorophenyl, Furfurylidene, m.  $222^{\circ}$ .<sup>19</sup>  
     Thenylidene, m.  $252^{\circ}$ .<sup>19</sup>  
     *p*-Chlorobenzylidene, m.  $201.5-204^{\circ}$ .<sup>148.5</sup>  
*o*-Tolyl, Benzylidene, m.  $171^{\circ}$ .<sup>5.5</sup>  
     Nitrobenzylidene, *m*, m.  $193^{\circ}$ ; *p*, m.  $240-60^{\circ}$ .<sup>165.5</sup>  
     Salicylidene, m.  $158^{\circ}$ .<sup>5.5</sup>  
     Anisylidene, m.  $208-12^{\circ}$ .<sup>165.5</sup>  
     Vanillylidene, m.  $168^{\circ}$ .<sup>165.5</sup>  
     Cinnamylidene, m.  $175^{\circ}$ .<sup>165.5</sup>  
*m*-Tolyl, Benzylidene, m.  $124^{\circ}$ .<sup>5</sup>  
     Nitrobenzylidene, *m*, m. about  $234^{\circ}$ ; <sup>5</sup> *p*, m.  $201^{\circ}$ .<sup>165.5</sup>  
     *o*-MeCOOC<sub>6</sub>H<sub>4</sub>CH:, m.  $231^{\circ}$ .<sup>5</sup>  
     Cinnamylidene, m.  $146^{\circ}$ .<sup>5</sup>  
*p*-Tolyl, Biphenylmethylene, m.  $170^{\circ}$ .<sup>145</sup>  
     Isopropylidene, m.  $180^{\circ}$ .<sup>145</sup>  
     Isobutylidene, m.  $175^{\circ}$ .<sup>145</sup>  
     Isoamylidene, m.  $177^{\circ}$ .<sup>145</sup>  
      $\alpha,\beta$ -Dimethylpropylidene, m.  $185^{\circ}$ .<sup>145</sup>  
     Cyclohexylidene, m.  $165^{\circ}$ .<sup>145</sup>  
     Methylcyclohexylidene, 3-, m.  $183^{\circ}$ ; 4-, m.  $163^{\circ}$ .<sup>145</sup>  
     Furfurylidene, m.  $200^{\circ}$ .<sup>145</sup>  
     Benzylidene, m.  $187.5^{\circ}$ ,<sup>5.5</sup>  $185^{\circ}$ .<sup>145</sup>  
     Hydroxybenzylidene, *o*, m.  $208^{\circ}$ ; *p*, m.  $257^{\circ}$ .<sup>145</sup>  
     Anisylidene, m.  $170^{\circ}$ ,<sup>165.5</sup>  $155^{\circ}$ .<sup>145</sup>  
     Vanillylidene, m.  $205^{\circ}$ ,<sup>145</sup>  $202^{\circ}$ .<sup>165.5</sup>  
     *p*-MeCOOC<sub>6</sub>H<sub>4</sub>CH:, m.  $198^{\circ}$ .<sup>5.5</sup>  
     Nitrobenzylidene, *o*, m.  $180^{\circ}$ ; *m*, m.  $212^{\circ}$ ; *p*, m.  $270^{\circ}$ .<sup>145</sup>  
     Cinnamylidene, m.  $185^{\circ}$ ,<sup>165.5</sup>  $180^{\circ}$ .<sup>145</sup>  
 Benzyl, Benzylidene, m.  $219^{\circ}$ .<sup>5</sup>  
 2,4-Dimethylphenyl, Benzylidene, m.  $171^{\circ}$ .<sup>5.5</sup>  
 2,4,5-Trimethylphenyl, Benzylidene, m.  $127^{\circ}$ .<sup>103</sup>  
     Anisylidene, m.  $174^{\circ}$ .<sup>103</sup>  
     *m*-Nitrobenzylidene, m.  $224^{\circ}$ .<sup>103</sup>  
 Naphthyl, Benzylidene,  $\alpha$ , m.  $159^{\circ}$ ;  $\beta$ , m.  $202^{\circ}$ .<sup>189.5</sup>  
     2,4-Dihydroxybenzylidene,  $\beta$ , m.  $190-200^{\circ}$ .<sup>5</sup>  
 2-Pyridyl, 1-Ethoxyethylidene, m.  $138^{\circ}$ .<sup>111</sup>  
 6-Methyl-2-pyridyl, 1-Ethoxyethylidene, m.  $166^{\circ}$ .<sup>111</sup>  
 2-Benzothiazolylmethyl, Benzylidene, m.  $219^{\circ}$ .<sup>201</sup>  
     Furfurylidene, m.  $229^{\circ}$ .<sup>201</sup>  
     Thenylidene, m.  $252^{\circ}$ .<sup>201</sup>

- 2-Methyl-5-benzothiazolyl, Thenylidene, m.  $236^{\circ}$ .<sup>201</sup>  
 2-Methyl-6-benzothiazolyl, Benzylidene, m.  $218^{\circ}$ .<sup>201</sup>  
     Furfurylidene, m.  $255^{\circ}$ .<sup>201</sup>  
     Thenylidene, m.  $259^{\circ}$ .<sup>201</sup>  
 Amino, Isopropylidene, m.  $149^{\circ}$ .<sup>158</sup>  
     4-Carboxy-2-butylidene, m.  $159.5^{\circ}$ .<sup>158</sup>  
     Benzylidene, m.  $199^{\circ}$ .<sup>158</sup>  $197^{\circ}$ .<sup>186</sup>  
 Acetamido, Benzylidene, m.  $205.5^{\circ}$ .<sup>186</sup>  
 Diacetyl-amino, Acetylaminomethylene, m.  $206^{\circ}$ .<sup>108</sup>  
 Benzylidene-amino, Isopropylidene, m.  $105^{\circ}$ .<sup>158</sup>  
*p*-Dimethylaminobenzylidene-amino, *p*-Dimethylaminobenzylidene, m.  $273^{\circ}$ .<sup>186</sup>  
 Vanillylidene-amino, Benzylidene, m.  $211^{\circ}$ .<sup>158</sup>  
 Anilino, Benzylidene, m.  $195^{\circ}$ .<sup>5</sup>  
     *o*-MeCOOC<sub>6</sub>H<sub>4</sub>CH:, m.  $170-3^{\circ}$ .<sup>5</sup>

### 3,5,5-TRISUBSTITUTED RHODANINES

- 3-Ethyl-5,5-dimethyl, oil,  $b_{10}$   $122-4^{\circ}$ .<sup>16.5</sup>  
 3-Ethyl-5-methyl-5-carboxymethyl, D and L, m.  $73.5^{\circ}$  (1H<sub>2</sub>O);  
     L anhyd., m.  $107-9^{\circ}$ ; inactive, m.  $108-11^{\circ}$ .<sup>102</sup>  
 3-Phenyl-5,5-dimethyl, m.  $116^{\circ}$ .<sup>16.5</sup>

### DIRHODANINES

- 3,3'-Ethylene-*bis*-rhodanine, m.  $194^{\circ}$ .<sup>135.5</sup>  
 3,3'-Ethylene-*bis*-rhodanine, 5-Benzylidene, m. about  $265^{\circ}$ .<sup>135.5</sup>  
     5-Cinnamylidene, m.  $235^{\circ}$ .<sup>135.5</sup>  
 3,3'-Ethylene-*bis*-5-ethoxymethylene rhodanine, m.  $207-9^{\circ}$ .<sup>124</sup>  
 3,3'-(1,2-Propylene)-*bis*-rhodanine, m.  $162-5^{\circ}$ .<sup>148.5</sup>  
 1,4-Phenylene-di(5-methylene) rhodanine, m.  $>360^{\circ}$ .<sup>2</sup>  
 1,4-Phenylene-di(3-ethyl-5-methylene) rhodanine, m.  $296^{\circ}$ .<sup>2</sup>  
 1,4-Phenylene-di(3-phenyl-5-methylene) rhodanine, m.  $>360^{\circ}$ .<sup>2</sup>  
 2,6-(1-Azaphenylene)-di(5-methylene) rhodanine, m.  $>360^{\circ}$ .<sup>2</sup>  
 2,6-(1-Azaphenylene)-di(3-phenyl-5-methylene) rhodanine, m.  $336^{\circ}$  dec.<sup>2</sup>  
 2,6-(1-Azaphenylene)-di(3-ethyl-5-methylene) rhodanine, m.  $229^{\circ}$ .<sup>2</sup>  
 5,5'-(5-Nitroisophthaloylidene) dirhodanine, m.  $>300^{\circ}$ .<sup>96</sup>

### MISCELLANEOUS

- 2-Amino-5-methyl rhodanine, m.  $65.6^{\circ}$ .<sup>158</sup>

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## CHAPTER 7

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# Carbon Disulfide

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The preparation of carbon disulfide by W. A. Lampadius in 1796 was a milestone in the history of chemistry. True, carbon disulfide is not a natural product, produced under the mysterious influence of the hypothetical *vis viva* but in physical properties it would be classed with ethanol rather than with minerals and metals. It has been found, however, in petroleum.<sup>83.5</sup> Actually, the isomerization of ammonium cyanate into urea by Wöhler was hardly a total synthesis from the elements, because in 1832 both ammonia and potassium cyanide were produced from organic materials. Whether or not carbon disulfide is an organic compound, it ranks with ethanol and benzene as the starting material for the synthesis of a vast number of organic compounds.

The great bulk of the dithioacids and esters, dithiocarbonates, and dithiocarbamates of Volume IV can be derived from it. The same may be said of the thioureas and derivatives that make up this volume, and of the thiocyanates of Volume VI. Reference must be made to these volumes for details of its reactions, the chief of which are given here only in outline.

Carbon disulfide is a volatile highly refractive liquid, freezing at  $-111.85^\circ$ , boiling at  $46.26^\circ$ , and having the density 1.2628, 20/4. Only a few of the many studies of its structure are given here. Carbon disulfide is linear<sup>77</sup> as shown by electron diffraction<sup>44, 204b</sup> and by use of the rotating sector.<sup>68</sup> The ultra-

422

violet absorption agrees with the structure  $\text{S}=\text{C}=\text{S}$ .<sup>136</sup> The C—S bond distance is 1.54Å,<sup>44</sup> 1.5532Å,<sup>83</sup> 1.56Å,<sup>68</sup> and the S—S bond distance is 3.16Å<sup>204b</sup> and 3.20Å.<sup>204a</sup> The structure of liquid carbon disulfide has been studied by X-rays.<sup>122</sup>  $\text{CS}_2$ ,  $\text{CO}_2$ , and  $\text{NO}_2^+$  are said to be isosters.<sup>155</sup>

### Manufacture

Except to say that the manufacture of carbon disulfide is conducted on a large scale in several ways, it will not be considered here. The union of carbon and sulfur is an equilibrium reaction; it is formed and decomposed at the same temperature<sup>12,5</sup> and is expressed by the following equation:<sup>29,5, 100, 177</sup>



Data are presented for several temperatures for the isotopic exchange of  $\text{C}^{14}$  with the carbon of carbon disulfide.<sup>181</sup> There is no interchange between radioactive sulfur and carbon disulfide below 100°,<sup>40</sup> but it does take place after 30–60 minutes at 257°.<sup>218</sup>

### Reactions

Carbon disulfide is decomposed by sunlight,<sup>128,5, 170,5</sup> by arc-light,<sup>53,5, 123,7</sup> and by silent electric discharge.<sup>127</sup> When strongly irradiated, carbon disulfide deposits a solid of the composition  $\text{CS}$ .<sup>119, 144</sup> Under ultraviolet light sulfur separates.<sup>23</sup> Irradiation gives free radicals even at temperatures of liquid nitrogen;<sup>143</sup> they are formed also by gamma radiation.<sup>156</sup> In sunlight, carbon disulfide and mercuric acetate give  $\text{S}(\text{HgOAc})_2$ , carbon oxysulfide, and acetic acid.<sup>12</sup> Only at room temperature does carbon disulfide react with  $\text{NO}_2$  and under the influence of ultraviolet radiation.<sup>87</sup>

Most of its reactions with other compounds may be regarded as additions to one or both of the double bonds. Hydrogen sulfide is produced when steam and carbon disulfide are passed over suitable catalysts at 400° and above.<sup>28</sup> A study has been made of the thermodynamics of this reaction.<sup>73</sup> This may be considered as the addition of two molecules of water across the double bonds. Carbon disulfide in hydrocarbon gases may be preferentially oxidised over a nickel sulfide catalyst at 140°.<sup>81, 217</sup>

## CHLORINATION

Early it was found that when chlorine and carbon disulfide are passed through a hot tube carbon tetrachloride and sulfur chloride are formed.<sup>118.5</sup> Aluminum chloride,<sup>118.5</sup> molybdenum-,<sup>4.5</sup> and antimony<sup>95.5b</sup> pentachlorides were shown to have a catalytic effect on the reaction.

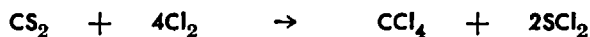
In the chlorination of carbon disulfide the first reaction is probably the addition of chlorine to one of the double bonds:



The thiophosgene which cannot be isolated takes up another molecule of chlorine:



This may be the chief product when iodine is used as a catalyst. In the presence of iron the chlorination goes further, the overall reaction being:



This is a commercial process for the manufacture of carbon tetrachloride; see volume I, p. 288.

## FLUORINATION

According to conditions, the fluorination of carbon disulfide gives a variety of products:  $\text{SF}_4$ ,<sup>171</sup>  $\text{F}_3\text{CSF}_5$ ,  $\text{SF}_6$ ,  $\text{F}_2\text{C}(\text{SF}_3)_2$ ,  $\text{F}_2\text{C}(\text{SF}_5)_2$ ,<sup>37</sup>  $\text{F}_2\text{C}(\text{SF}_5)_2$ ,  $\text{F}_3\text{SCF}_2\text{SF}_5$ ,  $\text{F}_3\text{CSF}_3$ ,  $\text{S}_2\text{F}_{10}$ , and a variety of additional cleavage products.<sup>193</sup> With iodine pentafluoride many products are formed:  $(\text{F}_3\text{CS})_2$ ,  $\text{SF}_4$ ,  $(\text{F}_3\text{C})_2\text{S}_3$ , and  $\text{CF}_4$ .<sup>88</sup>

## HYDROGENATION

Hydrogen may be added to both of the double bonds: <sup>140</sup>



As will be mentioned under removal, catalytic hydrogenation converts all of the sulfur to hydrogen sulfide.

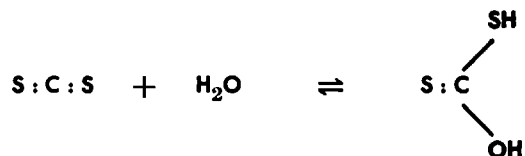
Carbon disulfide may be converted to trithioformaldehyde by treatment with zinc and hydrochloric acid.<sup>78.5</sup>

## WITH ALKALI

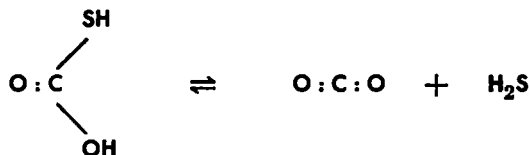
Carbon disulfide, like carbon dioxide, may be considered an acid anhydride. An aqueous solution of carbon dioxide is acidic:



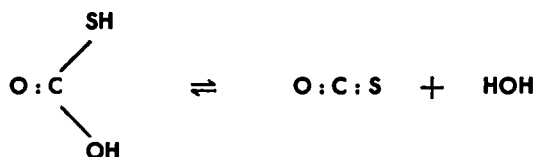
The analogous reaction with carbon disulfide may be written:



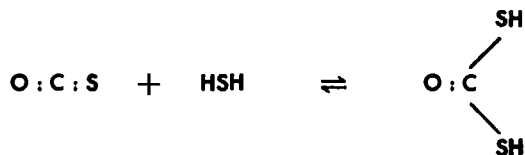
The mono-thiocarbonic acid may be in equilibrium with carbon dioxide and hydrogen disulfide



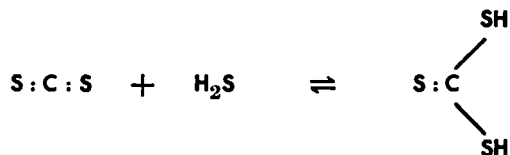
or with carbon oxysulfide and water



The addition of  $\text{H}_2\text{S}$  to carbon oxysulfide will give:



Carbon disulfide and hydrogen sulfide should give:



For simplicity, the compounds are written as molecules, though actually they would be dissociated. These are just some examples

of equilibria that can be written. As the solubility of carbon disulfide in water at 22° is only 2.18 g per liter, the amounts of the various compounds present would be extremely small. The addition of alkali would upset these equilibria. Actually, from carbon disulfide with two molecules of sodium hydroxide the final products are two molecules of sodium trithiocarbonate,  $\text{Na}_2\text{CS}_3$ , and one of sodium carbonate.<sup>33a, 198</sup>

There have been many studies of the kinetics of the reaction of carbon disulfide with sodium hydroxide. The velocity increases with the rate of agitation, showing, however, evidence of an approach to a limit. The temperature coefficient is about 8% per degree. The velocity increases with total volume of alkali, concentration of alkali, concentration of carbon disulfide in solvents, and quantity of carbon disulfide where no solvent is used.<sup>206b</sup> The nature of the solvent influences the rate. Addition of hydrogen peroxide increases the velocity by increasing the rate of diffusion into the aqueous phase, and it is proportional to the concentration if not excessive.<sup>72</sup> An equation has been given which shows the maximum reaction velocity in terms of the concentrations of sodium hydroxide and carbon disulfide.<sup>206b</sup> Assuming that sodium sulfide is an intermediate, an equation has been developed for calculating its concentration and that of sodium trithiocarbonate as functions of the reaction time between carbon disulfide and normal sodium hydroxide at 25°.<sup>206a</sup>

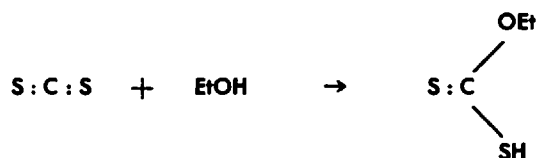
In a series of experiments with sodium hydroxide and carbon disulfide in a ratio of 2 : 1 the percentage of carbon disulfide converted to sodium sulfide reached a maximum of about 13% in 144 hours at 20°, 24 hours at 30°, and 8 hours at 40°. The conversion to sodium trithiocarbonate was practically complete in 240 hours at 20°, 48 hours at 30°, and 10 hours at 40°.<sup>33a</sup> Carbon disulfide reacts readily with sodium and potassium sulfides to give the trithiocarbonates.<sup>98, 195</sup>

### XANTHATES

The importance of carbon disulfide as a starting material has been pointed out. Synthetic organic chemistry of sulfur really commenced with the discovery of the xanthate reaction by Zeise in 1815, about the time that chemistry was beginning to take shape following the promulgation of the theories of Dalton. Zeise's synthesis of mercaptan came in 1834, just after Wöhler's

synthesis of urea. Had attention been given to Zeise's remarkably clear-cut experiments, the theories of organic chemistry would have advanced much more rapidly.

In this synthesis, the addition of water to carbon disulfide has been postulated as the first step in its reactions with sodium hydroxide. Alcohol may be supposed to add similarly:



In the presence of a base, to take care of the acid ester, the reaction will go to completion. It is simpler to write the reactions as molecular. The overall reaction is commonly written:



It should be noted that in the formation and decomposition of xanthates the oxygen of the alcohol remains attached to the alkyl. It took nearly one hundred years for chemists to realize that this is generally true in esterification. The addition of alcohol must proceed more rapidly than that of water. In one experiment, carbon disulfide was added to a solution of sodium hydroxide in 15% alcohol, in which there are fourteen times as many molecules of water as there are of ethanol. In spite of this, the yield of xanthate was 60%, from which we may guess that ethanol reacts over thirty times as rapidly as water.<sup>96b</sup>

Xanthation is a general reaction of the alcohols. Primary alcohols, even up to cetyl and octadecyl, react satisfactorily. Secondary alcohols are less active. Tertiary and diols, in which the hydroxyls are not separated by more than three carbon atoms, give poor results.<sup>188</sup>

At 60°, ethyl, isopropyl, and butyl ethers react with carbon disulfide and sodium hydroxide as would the corresponding alcohols.<sup>194</sup> The alkyl- and arylmercuric hydroxides with carbon disulfide and ethanol give xanthates  $\text{EtO}\cdot\text{CS}\cdot\text{SHgR}$ . Sodium hydroxide is a catalyst.<sup>121</sup>

#### XANTHATION OF CELLULOSE

The xanthation of cellulose, discovered in 1892 by Cross and Bevan,<sup>43</sup> has become of great industrial importance as the basis



for the manufacture of rayon and cellophane, two products which have achieved world-wide acceptance. Currently, some two billion pounds of cellulose are xanthated annually. For information on the process, reference should be made to the article by O'Shaughnessy in *Unit Processes*.<sup>148</sup> In brief, the cellulose is soaked in a concentrated solution of sodium hydroxide, and filter-pressed. The resulting alkali cellulose, as it is called, retains some water and unreacted alkali, and is treated in a rotating drum with carbon disulfide vapor. The product, which contains something like one xanthate group to two glucose units, is dispersed in water to form *viscose*, an amber colored liquid. After this has "ripened," it is filtered and extruded into dilute acid, usually sulfuric, to precipitate the cellulose xanthic acid which loses carbon disulfide to regenerate cellulose. For cellophane, the viscose is extruded as a sheet into the acid bath, from which it is passed through several washings and a dryer to be wound up on a roll. For rayon, the viscose is extruded through a spinaret that has eighty or more tiny holes, directly into an acid bath. The filaments unite to form a thread which is washed and wound on a cone.

This process, so simple in outline, has required the research of hundreds of chemists and the expenditure of millions of dollars to put it into commercial operation. The slightest variation in any step of the process shows up in the properties of the final product. In the first four decennial indices to *Chemical Abstracts* these are listed:

| No. | Articles | Patents | Books |
|-----|----------|---------|-------|
| 1.  | 13       | 56      | 1     |
| 2.  | 57       | 93      | 0     |
| 3.  | 326      | 450     | 4     |
| 4.  | 210      | 208     | 1     |

Several references are given to the recent study of the xanthation of cellulose.<sup>31, 33b, 35, 90, 184, 190</sup> There has been some interest in the xanthation of fiber<sup>89, 91, 153</sup> and of emulsion.<sup>33c</sup>

Polyvinyl alcohol is xanthated under the same conditions and to about the same degree as cellulose.<sup>212</sup>

## THIOXANTHATES

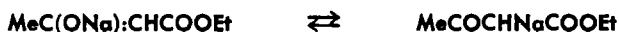
Thioxanthates, the esters of trithiocarbonic acid,  $\text{RS}\cdot\text{CS}\cdot\text{SH}$ , are formed readily from sodium or potassium mercaptides and carbon disulfide.<sup>32, 45, 96a</sup> Cuprous mercaptides react similarly with carbon disulfide.<sup>57</sup>

## WITH METALLO-ORGANICS

Carbon disulfide reacts readily with a Grignard reagent:



Metallo-organic compounds in general react similarly;<sup>168</sup> (see Volume IV, p. 38). The sodium derivative of acetoacetic ester is considered as an equilibrium mixture:

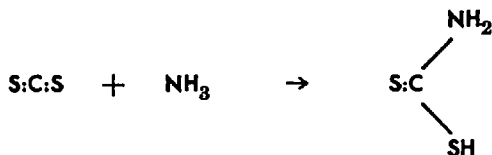


Assuming that the carbon disulfide reacts with the second form explains the fact that the products from acetoacetic ester and similar enolizable compounds are dithioesters, in which the dithioacid group is attached to the carbon atom rather than to the oxygen.<sup>107, 108, 109, 123, 201</sup>

Calcium cyanamide, water, and carbon disulfide give the salt of  $\text{N:C:N:C(SH)}_2$ .<sup>151</sup> Sodium cyanide and carbon disulfide in dimethylformamide give the compounds,  $\text{NC}\cdot\text{CS}_2\text{Na}\cdot 3\text{HCON-Me}_2$ .<sup>5</sup> Acetonitrile and carbon disulfide give the ester,  $\text{NC}\cdot\text{CS}_2\text{Me}$ .<sup>6</sup>

## WITH AMMONIA AND AMINES

As with water and with alcohol, the initial reaction with ammonia and amines may be assumed to be simple addition:



This is shown by the fact that when an active vinyl compound, or  $-\text{CH:CH}_2$ , is present direct addition takes place to form an ester:<sup>97</sup>



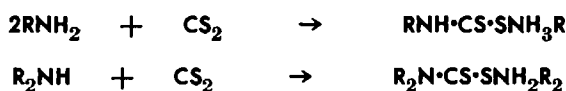
The second molecule of ammonia reacts with the acid to give the ammonium salt:



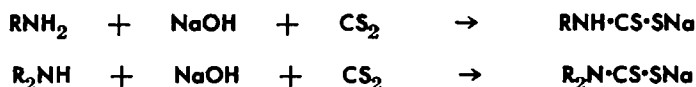
When the reactants are brought together in alcohol and ether is added, this salt crystallizes out in high yield.<sup>105</sup> It may decompose into  $\text{NH}_4\text{SCN}$  and hydrogen sulfide which combines with ammonia to give ammonium sulfide. With excess ammonia, ammonium trithiocarbonate and carbonate are formed.<sup>32.5</sup> At elevated temperatures— $150^\circ$  or above—ammonia and carbon disulfide will give thiourea and ammonium thiocyanate, probably an equilibrium mixture of the two.<sup>78, 118</sup>

The mechanism of the reaction has been studied.<sup>115, 116, 134</sup> Under certain conditions guanidine thiocyanate<sup>135</sup> and melamine may be produced.<sup>117</sup>

The reactions of primary and secondary amines with carbon disulfide are similar to those with ammonia: <sup>95.5a</sup>



Hydroaromatic and terpene amines react in the same manner.<sup>19.7</sup> It has been suggested that the second molecule of the primary amine may react with the sulfur of the CS group of the thio-carbamic acid.<sup>74</sup> Hydrogen peroxide facilitates the reaction between an aryl amine and carbon disulfide, but there has been some uncertainty as to the mechanism.<sup>19.5</sup> Sodium hydroxide can advantageously be substituted for the second molecule of the amine:



With two molecules of the amine the reaction may be only 95% complete, but with sodium hydroxide it is practically 100%.<sup>42</sup> The lead, copper, mercury, silver, gold, and platinum salts are readily formed whereas zinc, iron, manganese, cobalt, and nickel salts are not. These are precipitated from an aqueous solution and can be extracted with organic solvents. They are regarded as internal complexes. Some of them can be distilled in vacuum.<sup>51</sup>

Frequently the dithiocarbamate salts are made to undergo further reactions in the solutions in which they are formed. Thus,

substituted thioureas are obtained by refluxing higher amines, such as cetyl, with carbon disulfide in ethanol until no more hydrogen sulfide is evolved: <sup>129, 196</sup>



The elimination of hydrogen sulfide is facilitated by the presence of a heavy metal salt such as lead acetate.<sup>51</sup> A diarylthiourea is obtained directly from an aryl amine, carbon disulfide, and zinc oxide.<sup>75</sup>

Heating with a salt of a heavy metal causes the loss of hydrogen sulfide with the formation of an isothiocyanate: <sup>196</sup>



Carbon disulfide with dimethylamine monosulfide gives tetramethylthiuram disulfide.<sup>14</sup>

The reaction of carbon disulfide with aniline has been studied; <sup>186</sup> that with aromatic amines is influenced by the nature of the solvent.<sup>74, 133</sup>

Carbon disulfide reacts regularly with  $\alpha$ -aminoacids to form dithiocarbamates.<sup>183</sup> This reaction has been investigated polarographically <sup>213a, 216</sup> and kinetically.<sup>213c</sup> It takes place even with polypeptides and proteins <sup>124, 126</sup> and may be used for their systematic degradation. The resulting dithiocarbamates may be separated chromatographically <sup>215</sup> and the amino-acids regenerated from them.<sup>213b</sup> Glycine may be determined by polarographic titration of the dithiocarbamate.<sup>214</sup> Carbon disulfide may react with the  $-\text{NH}_2$  group of a sulfonamide.<sup>163</sup>

Two molecules of carbon disulfide, two of sodium hydroxide, and one of hydrazine form disodium hydrazine-*bis*-dithiocarbonate.<sup>207</sup> Under certain conditions a cyclic compound is said to be formed.<sup>25.5</sup> The hydrazide of a carboxylic acid reacts with carbon disulfide to give an oxadiazoline-5-thione.<sup>4</sup> From one molecule of carbon disulfide and one of a diamine an inner thio-carbamic salt may be obtained. By loss of hydrogen sulfide such salts may be converted to cyclic or to polymeric thioureas according to the length of the carbon chain in the diamine.<sup>54, 110, 176, 180, 197, 208</sup> 4,4'-Diaminothiocarbanilide has been obtained from *p*-phenylenediamine; <sup>15</sup> 4,4'-diaminophenylthiocarbanilide, from benzidine; <sup>162</sup> and sodium piperazinodithiocarbamate, from piperazine.<sup>47</sup>

## OTHER REACTIONS

Carbon disulfide forms addition products with tertiary phosphines.<sup>48, 101b, 104, 130, 131, 147, 178</sup> Structures of these have been suggested.<sup>49, 50, 101a</sup> A pure phosphine may be obtained by distilling a carbon disulfide compound from alcoholic sodium or potassium hydroxide.<sup>101a</sup> Two melting points,  $95^{\circ}$ <sup>203</sup> and  $122^{\circ}$ ,<sup>103</sup> have been given for the complex,  $\text{Et}_3\text{P}\cdot\text{CS}_2$ . Carbon disulfide also forms complexes with arsines and stannanes.<sup>104</sup>

A study has been made of the kinetics of the reaction of carbon disulfide with the azide ion to form  $\text{SCSN}_3^-$ . The reaction is of second order and the energy of formation is 21,100 cal/mole.<sup>94</sup> Some such reaction must account for the catalytic influence of carbon disulfide on the reaction between the nitride ion and iodine.<sup>22, 199</sup> Carbon disulfide as a solvent may influence the course of a Friedel-Crafts reaction.<sup>7, 59, 141, 157, 159</sup> This has been attributed to the low solubility of some of the aluminum chloride complexes in carbon disulfide.<sup>182</sup> The yield of benzoic acid in the oxidation of toluene over a carbon-silver catalyst is raised from less than 1% to 26% by the presence of carbon disulfide.<sup>55</sup> It cuts down the formation of by-products in the oxidation of methanol to formaldehyde.<sup>150</sup>

In the catalytic production of hydrogen cyanide from methane and ammonia, carbon disulfide increases the yield.<sup>120</sup> It activates the molybdenum, tungsten, or chromium catalysts in the hydrogenation of aromatic nitro compounds.<sup>175</sup> It accelerates the solution of aluminum in hydrochloric acid<sup>154</sup> and facilitates the formation of the Grignard reagents.<sup>202</sup> Carbon disulfide is an initiator in dust explosions on account of its low ignition temperature.<sup>60</sup> It retards polymerization of isoprene and of styrene.<sup>56</sup> A mixture of carbon disulfide and hydrogen sulfide passed over alumina at  $100\text{--}160^{\circ}$  gives 66–80% methyl mercaptan, the balance being mainly methyl sulfide.<sup>9</sup>

## Detection and Determination

As a large part of the 500 million pounds of carbon disulfide required for the manufacture of 2 billion pounds of cellophane and rayon annually escapes ultimately into the atmosphere, methods of detection and estimation are important. It may be also a hazard in grain storage and is an objectionable impurity

in coal-tar benzene and in other light hydrocarbons. The methods for determining carbon disulfide in hydrocarbons have been reviewed.<sup>36</sup>

In the following brief listing, articles appearing before Volume 40 of *Chemical Abstracts* are not included, because information of value has reappeared in later articles.

The ready conversion of carbon disulfide to xanthates is the basis for a group of methods.<sup>1, 84</sup> The xanthate may be estimated by titration with iodine,<sup>71, 86, 95</sup> or by conversion to its copper,<sup>46, 69a, 69b, 71</sup> mercury,<sup>69a</sup> or molybdenum derivatives.<sup>10, 219</sup>

The dithiocarbamic acid, from the reaction of carbon disulfide with diethylamine, gives a characteristic copper salt<sup>11, 21, 24, 52, 64, 189</sup> which may be determined colorimetrically;<sup>53, 79, 111, 209</sup> or the acid can be determined by potentiometric titration,<sup>161a</sup> or by anodic wave.<sup>220</sup> By the aid of diethylamine it is possible to determine carbon disulfide down to 0.6 mg in one cubic meter of air.<sup>173</sup> Piperidine<sup>19, 112, 132</sup> and morpholine<sup>39, 161b</sup> may be used in the place of diethylamine. Carbon disulfide can be detected with piperazine<sup>17</sup> and with copper tetramine sulfate.<sup>29</sup>

The hydrogen sulfide from its catalytic hydrogenation may be estimated.<sup>67</sup>

The limit of accuracy by ultraviolet absorption<sup>2, 179</sup> is 0.01 mg per liter of air<sup>18</sup> and 7.5 parts per million.<sup>191</sup> Dependence is put on the characteristic 4.55 micron band.<sup>66a</sup> It may also be determined by its infrared absorption.<sup>26, 66b, 80, 106</sup>

Other methods depend on oscilloscope;<sup>92a, 92b</sup> an "electronic polariscope";<sup>92c</sup> chromatography, using a Fractovap apparatus;<sup>194.5</sup> refractivity;<sup>125</sup> theory of formaldehyde catalysis;<sup>169</sup> titration of carbon disulfide in pyridine solution;<sup>46.5</sup> electro-metric titration of the sulfate ion;<sup>99</sup> and an oil extraction.<sup>128</sup>

### Removal of Carbon Disulfide

The methods of removal of carbon disulfide from air<sup>185</sup> and from coal gas<sup>61</sup> have been reviewed.

Carbon disulfide may be removed from gases by oil scrubbing.<sup>41, 152</sup> Considerable attention has been given to the removal of carbon disulfide from benzene made from sulfur-containing coal. It may be taken out of crude benzene and other hydrocarbons by conversion to xanthate by treatment with alcoholic sodium hydroxide.<sup>82, 93, 142, 205</sup> Alkali-cellulose has been recom-

mended for use with gases.<sup>3, 113, 114</sup> From gases or liquids, such as crude benzene, it may be extracted by conversion to a thio-carbamate by treatment with ammonia, with<sup>174</sup> or without<sup>8, 172</sup> sodium hydroxide, or with an amine,<sup>58, 76, 146, 170, 200, 211</sup> to which may be added sodium hydroxide<sup>149, 160</sup> or a metal oxide.<sup>137</sup> Other agents in a great variety have been recommended for removal of carbon disulfide: water<sup>16</sup> or sodium hydroxide<sup>76.5</sup> at an elevated temperature; sulfuric acid;<sup>158, 184</sup> calcium,<sup>34</sup> copper,<sup>144.5</sup> or iron oxides;<sup>145</sup> alkali sulfide,<sup>166</sup> ammonium<sup>20, 85, 210</sup> or sodium polysulfides;<sup>38</sup> and chlorous acid.<sup>167</sup> Radioactive isotopes have been employed in the study of removal of carbon disulfide.<sup>25</sup> The carbon disulfide, in a gas containing free hydrogen, is hydrogenated to hydrogen sulfide by passing it over a nickel catalyst at 400°.<sup>13, 27, 63, 192</sup> In a study of this reaction it was concluded that the active catalyst is a labile nickel sulfide,  $\text{Ni}_3\text{S}_2$ .<sup>62</sup> Other catalysts are copper thiomolybdate,<sup>187</sup> cobalt sulfide,<sup>70</sup> iron-chromium-,<sup>102</sup> or molybdenum sulfides.<sup>30, 70</sup>

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